Corrosion

Official Publication
NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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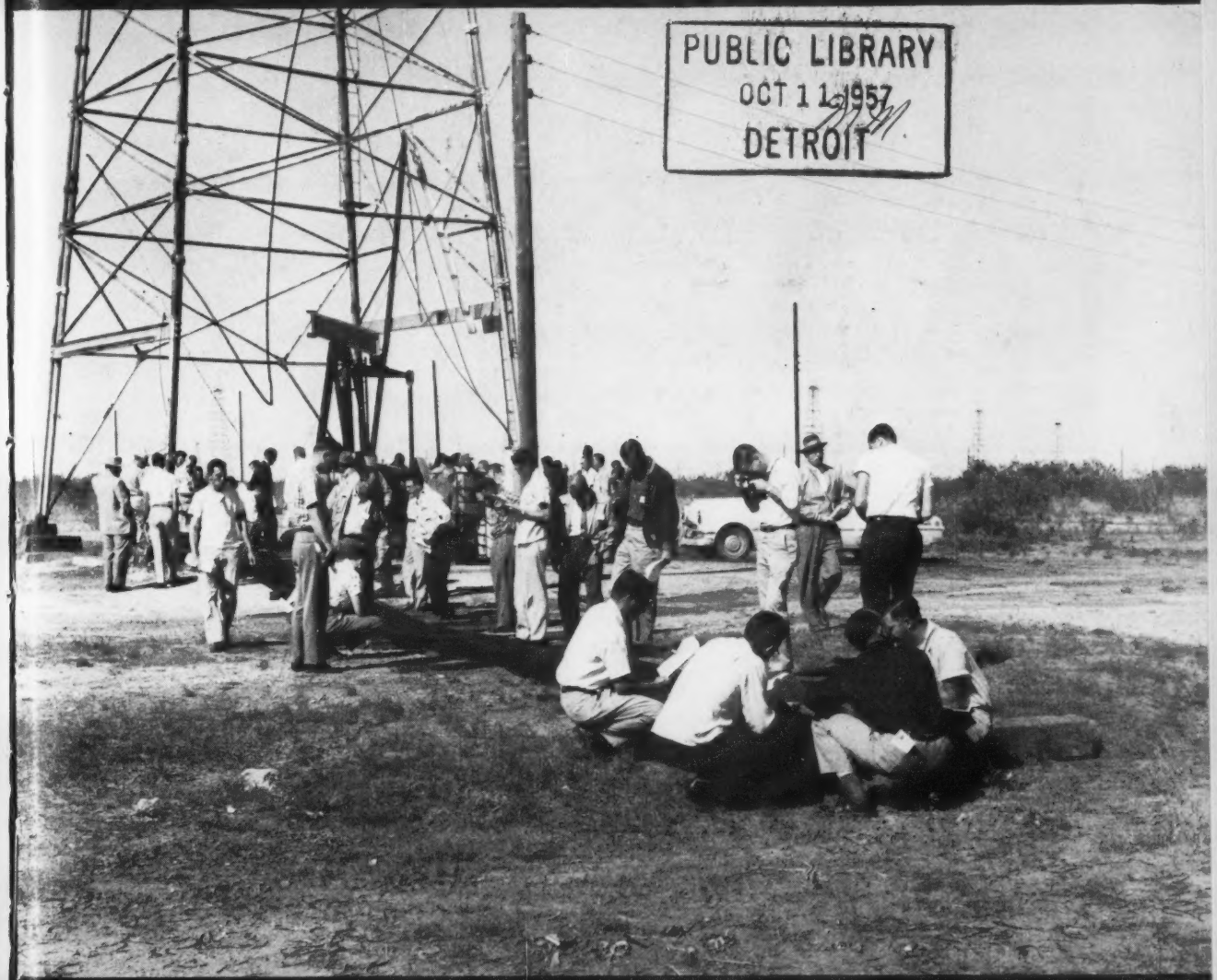

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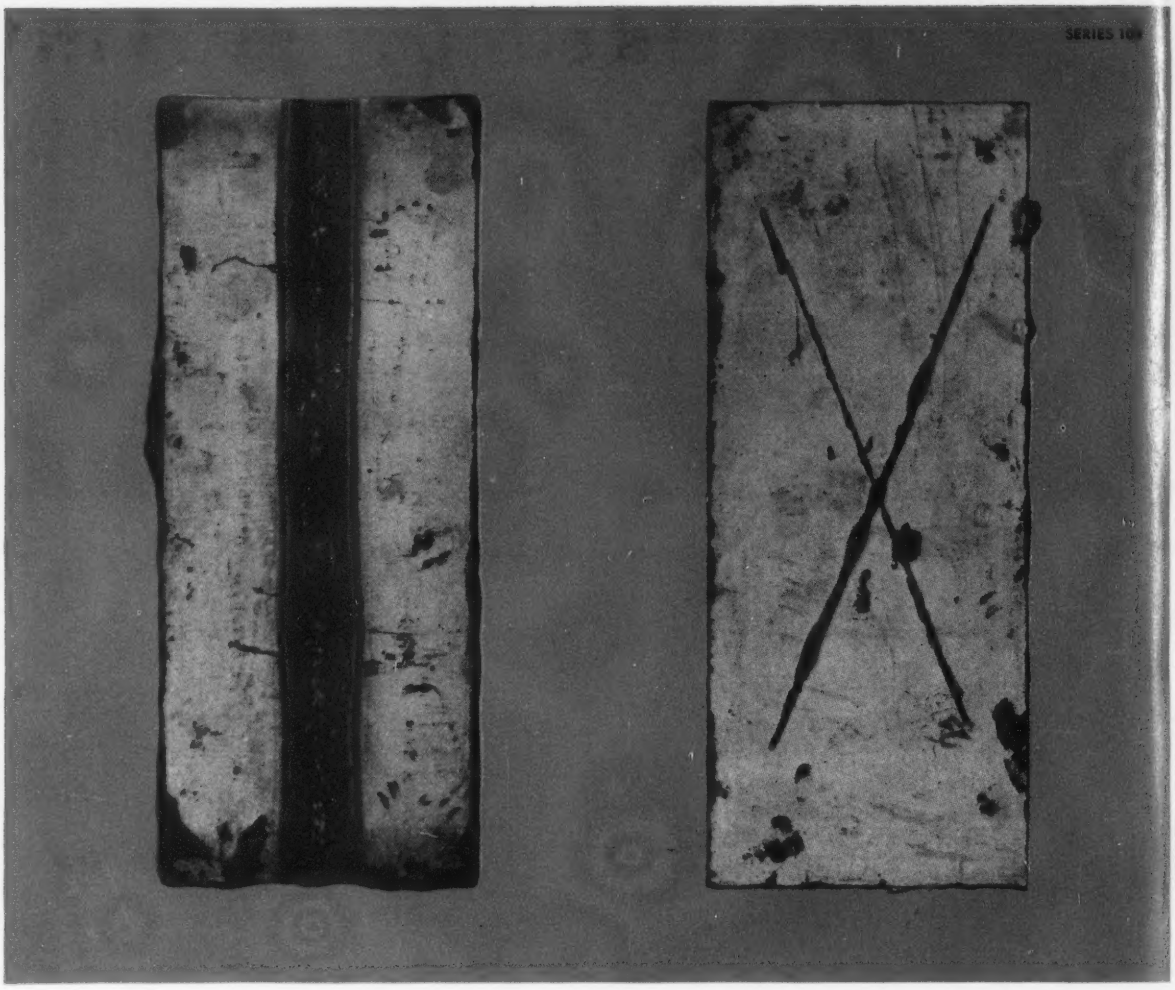

CORROSION'S FOCAL POINTS are easy to detect with a new Amercoat testing method using iron and caustic indicators in a saline gelatin bath. On the welded and scored steel panels shown above after test, unworked surface areas turned red to indicate cathodic properties; but the weld, scores and edges turned blue (here shown blackened) to reveal themselves as anodic corrosion breeders. Rivets, threads, crevices and abrasions also show anodic under test. Unless such areas are effectively sealed from moisture, oxygen and ions they will erupt as focal points of corrosion cells.

How to protect "trouble spots" against corrosion

Ask a corrosion engineer where metal corrosion is likely to attack first. Chances are that he will name rivets, threads, sharp edges, angles, crevices and welds. These are the areas of stress concentration, work hardness, fissures and abrasions. They tend to be anodic and actually breed corrosion. And although they warrant increased protection, conventional coatings pull away from their sharp profiles and leave them inadequately covered.

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devoted entirely to corrosion research and control

Published monthly as its official journal, by the National Association of Corrosion Engineers, Inc., at Houston, Texas, U. S. A., as a permanent record of progress in corrosion control.

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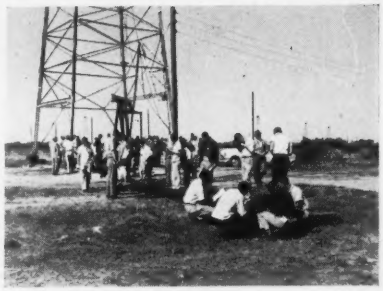

THIS MONTH'S COVER—Permian Basin Corrosion Tour members are shown here during the 1955 tour inspecting sucker rods pulled from a producing well. The Oct. 23-25 Seventh Biennial Tour sponsored by NACE's Permian Basin Section features numerous similar inspection trips in air conditioned busses. Over 300 are expected to register. Tour headquarters is Lincoln Hotel, Odessa.

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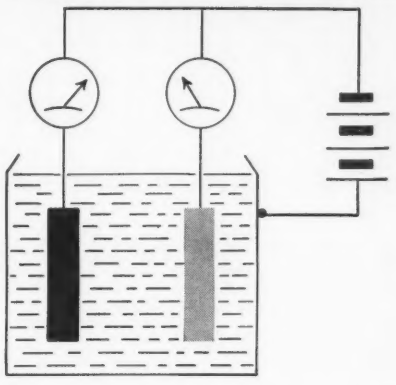

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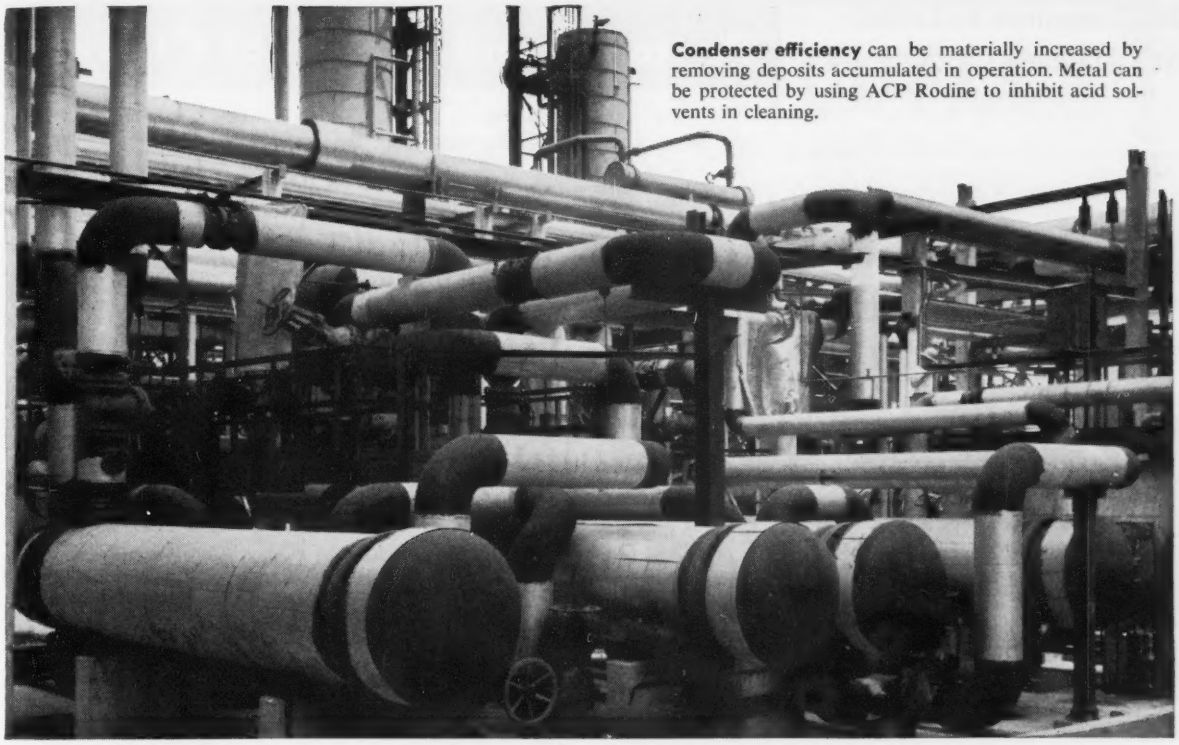
(Continued on Page 10)



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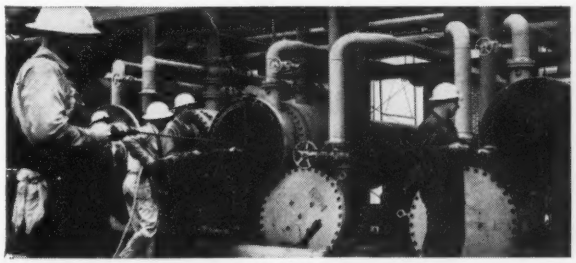

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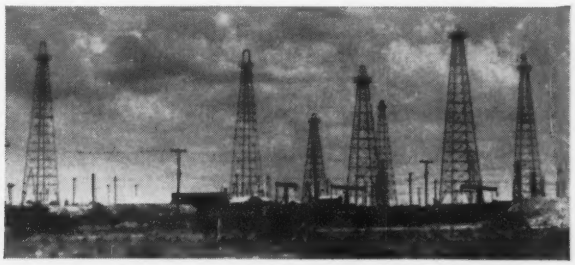

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New Chemical Horizons for Industry and Agriculture

36 Companies to Exhibit At Oklahoma City Oct. 1-3

The South Central Region Exhibition at the Oklahoma City Municipal Auditorium October 1-3 will feature the products and services of 36 companies. This makes it approximately equal in size to the regional exhibition held at San Antonio in 1956.

In addition to those listed in earlier issues of CORROSION, the following firms exhibited:

Brance-Krachy Company, Inc., Houston, Texas
Cook Paint & Varnish Company, Kansas City, Missouri
The Holcombe Company, Shreveport, Louisiana
South Central Region of NACE
Southwestern Plastic Pipe Company, Mineral Wells, Texas

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(Continued From Page 8)

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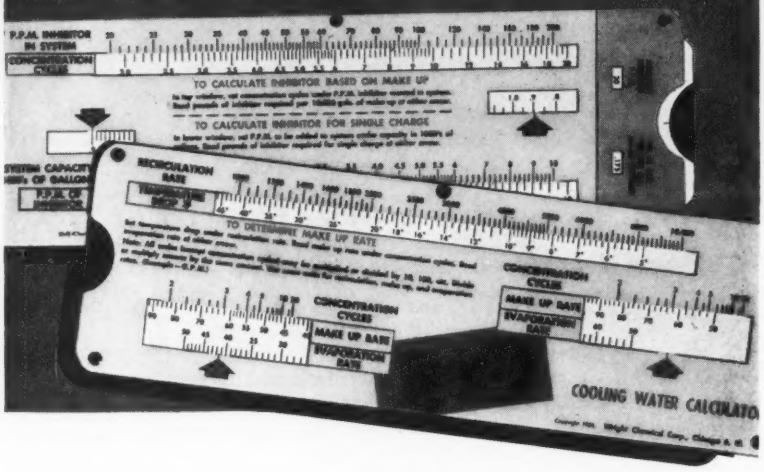
Announcing the New WRIGHT Cooling Water Calculator



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National Chemical Show Scheduled at Chicago

The Tenth National Chemical Exposition, sponsored by Chicago Section, American Chemical Society will be held September 9-12, 1958 at the International Amphitheater in Chicago. This coincides with the 134th national meeting of the American Chemical Society there.

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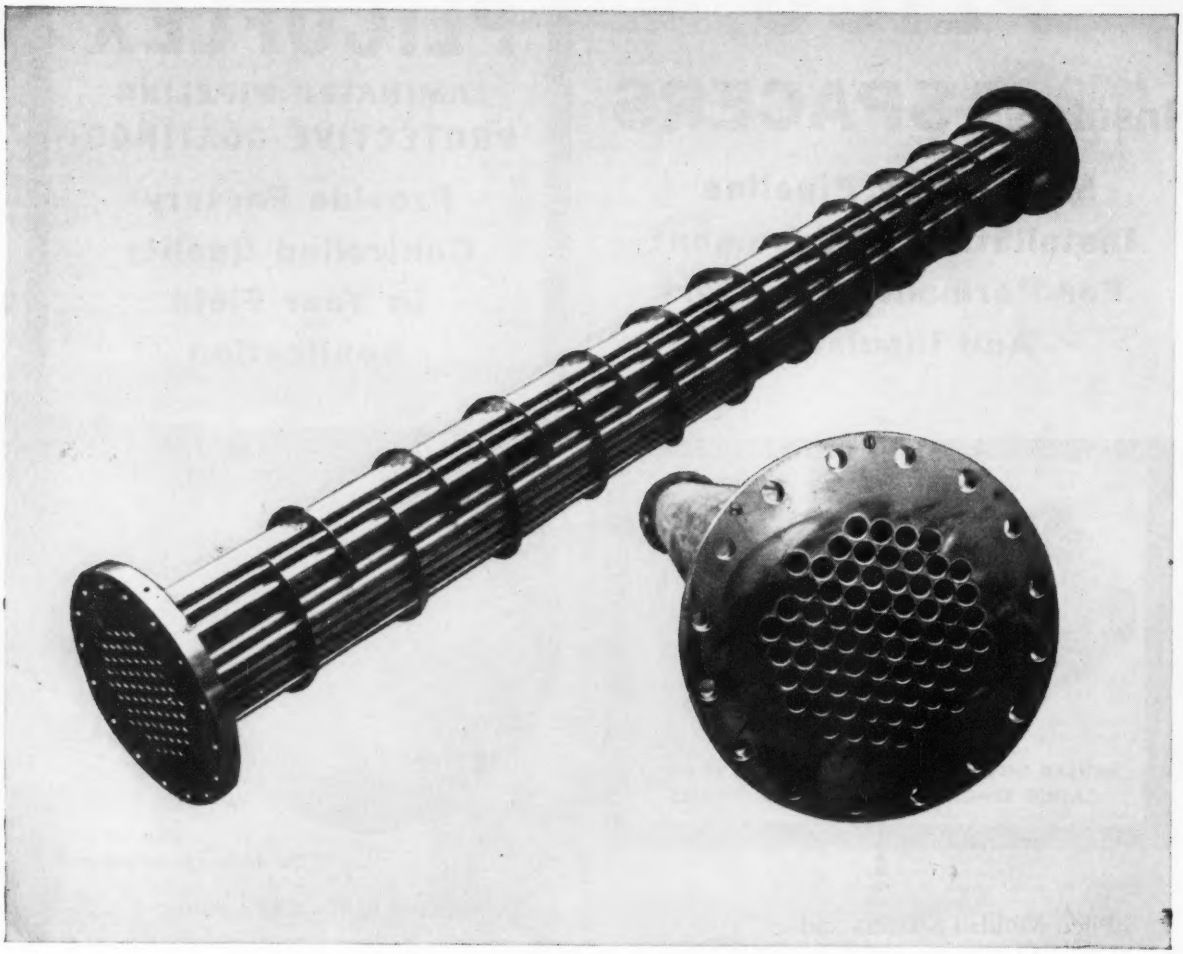

• *The life of this titanium 70-tube condenser assembly, now handling 60% nitric acid at 195° C. and 180 psi, is estimated to exceed 10 years.*

Titanium condenser tubes expected to outlast stainless 30 to 1 handling nitric acid

Corrosive vapors of 60% nitric acid gnawed through an average of three stainless-steel tube bundles a year in a chemical firm's condenser. Maintenance and replacement costs were running as high as $18,000 a year. Then titanium tubes were installed. Based on a service life of 10 years, titanium will save at least $162,000 in maintenance and replacement costs.

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In your process-development work, take full advantage of the corrosion and cavitation resistance, light weight and high strength of titanium . . . important considerations for continuous flow, high temperature and pressure processes. For further information, get in touch with Du Pont. And be sure to mail coupon below for an informative booklet about titanium—its properties and applications. E. I. du Pont de Nemours & Co. (Inc.), Pigments Department, Titanium Market Development Section, Wilmington 98, Del.

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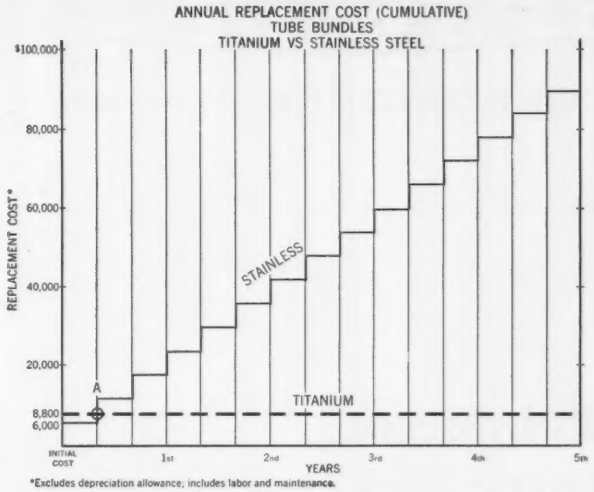
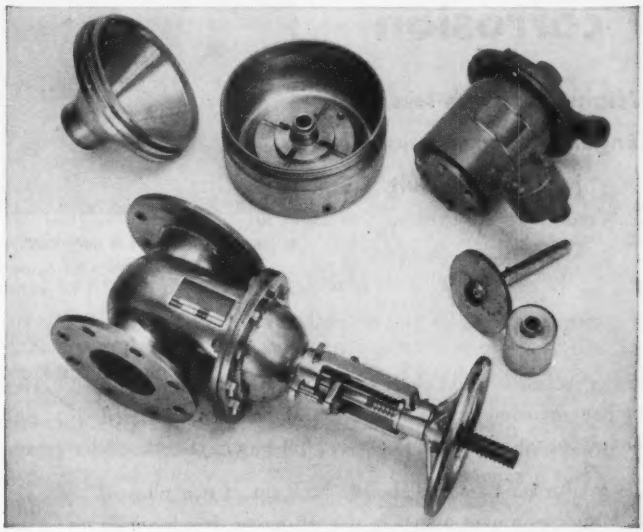
BETTER THINGS FOR BETTER LIVING . . . *THROUGH CHEMISTRY*



INITIAL AND ANNUAL REPLACEMENT COSTS of titanium and stainless tube bundles are compared above. Initial cost of the titanium tube bundle, shown at left, is $8,800, vs. $6,000 for stainless. But note how the $2,800 difference is absorbed within 4 months of service (Point A). Savings are approximately $81,000 after 5 years . . . $162,000 after 10 years, the expected service life of titanium tubes.



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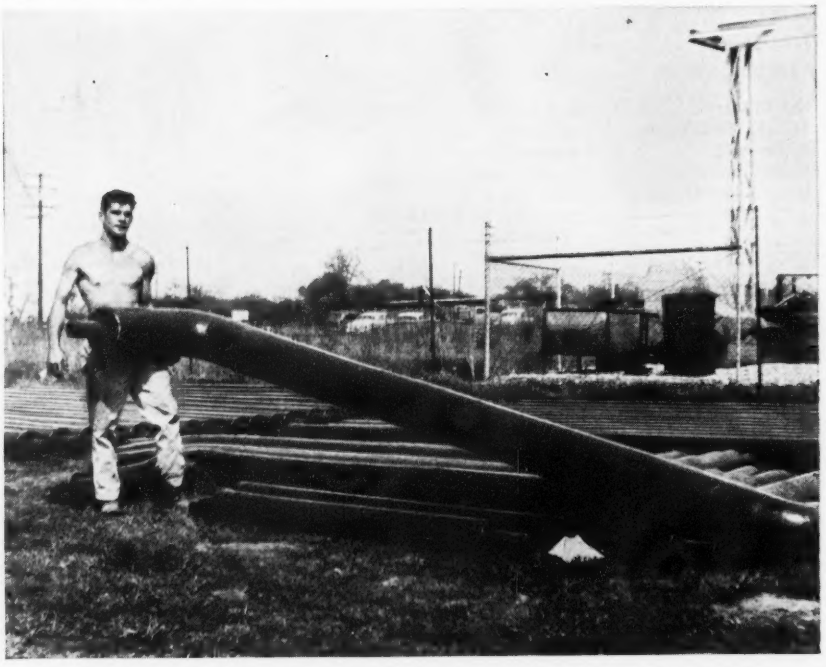

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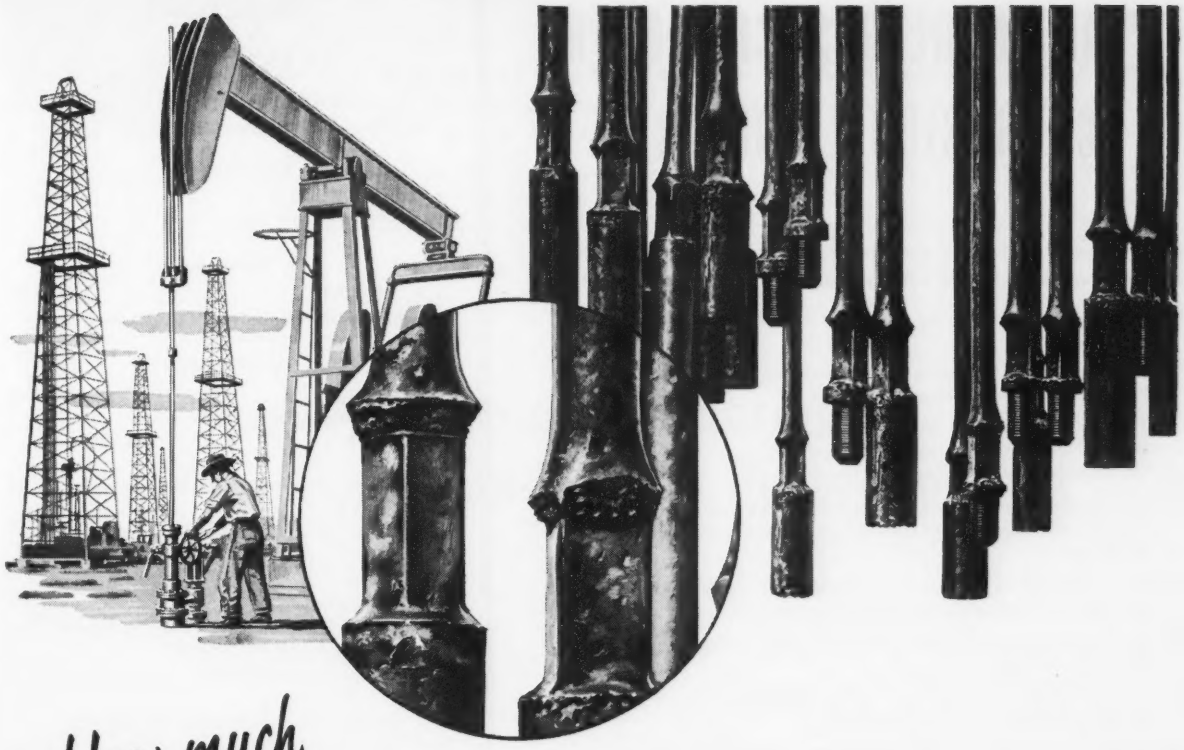

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(a) To promote the prevention of corrosion, thereby curtailing economic waste and conserving natural resources.

(a) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

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(g) To foster cooperation between individual operators of metallic plant and structures in the joint solution of common corrosion problems.

(h) To invite a wide diversity of memberships, thereby insuring reciprocal benefits between industries and governmental groups as well as between individuals and corporations.

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Corrosion

OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 13 October, 1957 No. 10

Topic of the Month

Epoxy-Fiberglass Armoring For Corrosion-Erosion Protection

By W. F. OXFORD, JR.*

THE USE of fiberglass epoxy armoring to prevent corrosion and erosion has come to the front in the oil production field. The uses to date have been in connection with (1) dual completion wells, (2) gas lift valve mandrels, and (3) splash zone protection on offshore structures.

Dual Completion Wells

In a dual string-dual completion well, part of the deeper producing string is in a critical position because it is exposed to the production and the erosive, corrosive effect of the flow from the upper producing zone.

To alleviate this condition, several operators have placed flow couplings, externally coated with lead, rubber, and other substances, in the vital area and have reported some failures within six months exposure.

It has been the practice of an oil company to combat this problem by externally coating flow couplings and tubing used in the critical area in the following manner:

1. One coat epoxy resin.
2. Three servings (half-lap) of 10 mil fiberglass cloth saturated with epoxy resin.
3. One finish coat epoxy resin. Upon completion, the epoxy-fiberglass coating is approximately 3/16 inch in thickness.

Figure 1 is a cross section of oil field tubing showing the thickness of the armoring.

After ten months exposure in two different wells, an inspection of the armored flow couplings revealed no damage.

Gas Lift Valve Mandrels

Gas lift valve mandrels in many cases are subject to destruction by erosion and/or corrosion if valve failure occurs. In order to prevent mandrel destruction, the armoring of the mandrels between the lugs has been a satisfactory procedure. The coating system for mandrel armoring is the same as that used in flow couplings in dual completion wells.

Figure 2 is a photograph of the epoxy resin-fiberglass armored mandrel.

Splash Zone Protection

Well guards protecting offshore wells are being successfully protected against corrosion by the epoxy resin-fiberglass armoring in the splash zone. The coating application is usually applied three feet above and two feet below Mean Gulf Level. This system is easily applied to the piping structure. The application price is approximately one-third that of armoring with some of the more corrosion-resistant metals.

Figure 3 is a photograph of a well guard which has been armored in the splash zone with the epoxy resin-fiberglass system.

* Sun Oil Company, Beaumont, Texas.

Figure 1—Typical cross section of oil field tubular goods showing epoxy resin-fiberglass armoring used on parallel string dual completions.

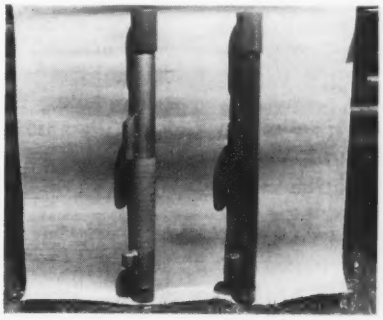

Figure 2—Gas lift valve mandrels with epoxy resin-fiberglass armoring to prevent erosion in the event of gas lift valve failure.

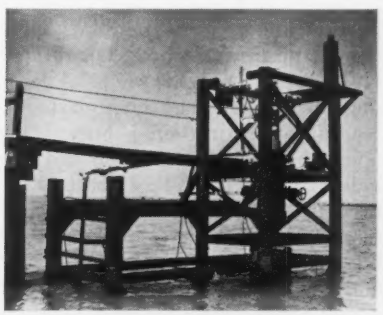

Figure 3—Splash zone protection utilizing epoxy resin-fiberglass armoring on offshore well guard.

Artificial Insulating Joints for Stray Current Mitigative Measurements*

By G. H. CANTWELL

Introduction

ONE OF THE mitigative measures which is often used in stray current corrosion work, either singularly or with other measures, is the insulating joint. It is a device for interrupting the flow of current on a metallic structure by inserting an insulator in the metallic path. Insulating joints are usually constructed in metallic sheathed cables by removing a small section of the sheath and replacing it with insulating tapes, and in metallic pipes by inserting an insulating gasket or ring between sections of the pipe.

The use of artificial insulating joints associated with the usual tests made prior to recommending permanent measures will eliminate the necessity of placing temporary insulating joints, or estimating the effect of an insulating joint. Also, since the equipment used for the artificial insulating joint is portable, the tests readily can be made from several locations.

Description

The principle of the artificial insulating joint is a variation of the application of a "Reduction Current Test" to an underground structure for predetermining the results of the application of cathodic protection. The use of "Reduction Currents" for testing purposes is described in other papers and in the Bell System Practices.

In order to simulate an insulating joint, the "Reduction Current" is applied twice, once for each structure on each side of the insulating joint. This is described in detail below.

Figure 1 shows a section of underground cable where other measurements and circumstances indicate it desirable to install an insulating joint at manhole "B." Currents "I_1" and "I_2" are the normal stray currents and are approximately equal, and the potential V is the potential to earth of the structure.

For the purpose of illustration, currents "I_1" and "I_2" are one and the same at manhole "B." Where the current is large enough to produce a sizeable reading of IR drop on a short section of cable sheath, point "b" (see Figure 2) should be moved from manhole "A" to "B." The Current "I_2" should be measured by conventional means prior to making these tests in order to determine the needed current carrying capacity for ammeter A, adjustable resistance R and the DC supply E (see Figure 2). The Current I_3 required to simulate an insulating joint will seldom be more than twice I_2. After the above has been determined and the equipment connected as shown in Figure 2, resistance R is adjusted until I_1 is zero as indicated by a zero reading on the millivolt meter. This condition has stopped the flow of current beyond manhole "B" such as an insulating joint would do, and has produced a change of potential to earth at manholes "B," "A," and on beyond. The potentials to earth at manhole "B" V_1 are those that would be found on the left side of the insulating joint when permanently placed.

Figure 1—Condition of the cable prior to simulating or placing an insulating joint.

G. H. CANTWELL has been associated with corrosion work for the Indiana Bell Telephone Company, Incorporated, Indianapolis, Indiana, for twenty years. He started as an electrolysis tester and was promoted in 1940 to Plant Engineering where supervision of electrolysis testing was one of his duties. He attended Purdue University, is a registered professional engineer in the state of Indiana, a member of NACE, and vice-chairman of NACE Task Group T-4B-6.

Abstract

In making electrical measurements pertinent to determining stray current mitigative measures, it is often desirable to determine the effect of a proposed insulating joint at a predetermined location. By applying a direct current source between the structure and the earth in the manner described in this paper, simulation of an insulating joint is achieved. Since the method is entirely electrical, the placing of temporary insulating joints for test purposes is eliminated. This method can be applied to any structure carrying stray DC earth currents.

In addition to simulating an insulating joint, the associated current and potential measurements are used to determine (1) the resistance to earth of the structure on each side of the point of application, (2) the potential to earth that would exist on each side of a permanent insulating joint, (3) the potential across the permanent insulating joint. They also provide data for determining the resistance and current carrying capacity for a bridging resistor. **4.5.3**

* Submitted for publication March 20, 1957. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

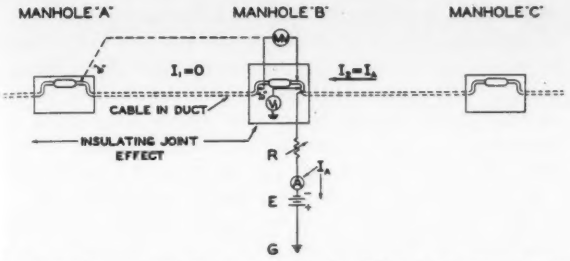

Figure 2—Arrangement for creating the insulating joint effect in the manhole "B" to manhole "A" direction.

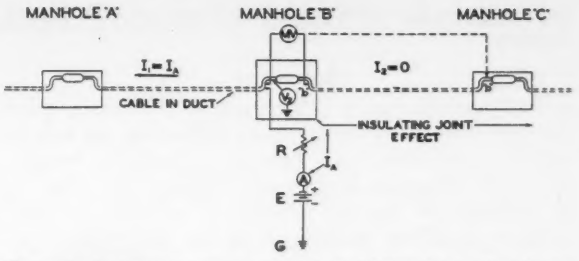

Figure 3—Arrangement for creating the insulating joint effect in the manhole "B" to manhole "C" direction.

In order to find out what happens to the currents in the manhole "B" to "C" direction, the polarity of the supply E is reversed (see Figure 3). Then the millivoltmeter is moved to indicate the current flow in the manhole "B" to "C" section and the adjustable resistance adjusted to stop Current I_2. The potentials at manhole "B" V_2 are those that would be found on the right side of the insulating joint when permanently placed, and those at manhole "C" and beyond will be those found with an insulating joint at manhole "B." Currents and potentials in the manhole "B" to "C" direction and beyond can be measured to determine the effect of an insulating joint at manhole "B."

When the stray current being blocked varies, the resistance R must be adjusted accordingly to keep a zero current in the section under investigation.

The ground point G, as required in "Reduction Current" tests, should be sufficiently removed from the structure to minimize its influence on the potentials to earth of the structure.

Determining the Resistance of the Structure

For the purposes of more clearly illustrating and analyzing the condition, assume that the insulating joint has been placed at manhole "B" and that potentials V_1 and V_2 are as determined by the artificial insulating joint test.

Viewing the cable sheath electrically from manhole "B" toward manhole "C" in respect to earth, the various potentials and resistances illustrated in Figure 4 can be considered as an electrical source with an open circuit potential V_2, since V_2 was determined with zero current flow, and an internal resistance equal to the summation of the resistances. The same consideration can be given to the cable sheath in the manhole "B" to "A" direction.

Since the open circuit potential is reduced by the IR drop within the source when a current flow exists, the internal resistance can be determined by dividing the change in potential by the associated change of current.

$$\frac{V - V_2}{I_2 - O} = \text{Int. Res. (Current flow for } V_2 \text{ is zero.)}$$

The internal resistance in the manhole "B" to manhole "A" direction can be determined in the same manner.

$$\frac{V - V_1}{I_1 - O} = \text{Int. Res. (Current flow for } V_1 \text{ is zero.)}$$

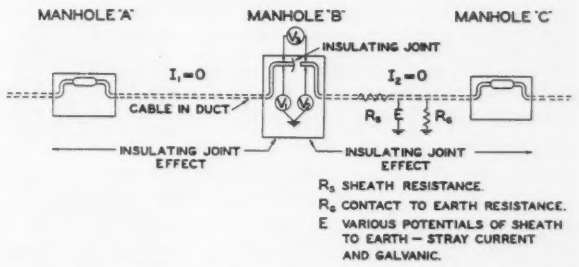

Figure 4—Conditions as a result of placing an insulating joint at manhole "B."

The potential across the insulating joint, as determined by triangulation, will be equal to the algebraic sum of V_1 and V_2.

The maximum current flow when the insulating joint is shunted with a low resistance bond should equal I_1 and is equal to the potential across the insulating joint divided by the sum of the internal resistances on each side of the insulating joint.

Application

The principal use of the artificial insulating joint has been to permit the study of the effects of insulating joints in aerial cables which have protective sheath grounding remote to an underground section under cathodic protection. By making these studies, it was frequently found that the grounding did not appreciably impair the cathodic protection and that it would be inadvisable to place an insulating joint. However, in other cases, the tests indicated the use of an insulating joint was advisable.

In making the above tests, potentials and currents on both sides of the proposed insulating joint were measured in order to determine the effect on the cathodic protection and on the resulting electrolysis condition which would result on aerial cable dips in the isolated section. These tests also afforded a means of determining the approximate grounding resistance on each side of the proposed insulating joint.

In one case an artificial insulating joint was used to remove a 200 milliampere current from a section of underground cable which had experienced several sheath failures within a few years. As a result, it was found that the removal of the current did not change the potential to earth of the cable sheath, and since no measurable loss of current had been detected in the section, it was concluded that this

current was not the principal cause of the sheath failures.

Summary

The installation of a permanent insulating joint verified the results obtained by following the recommended procedure for creating the artificial insulating joint. The accuracy of the results was found to be determined by the ability to maintain a zero current condition while making the tests.

Rapidly varying and reversing currents would complicate the application of an artificial insulating joint. For this reason, and also because an insulating joint is seldom required under these conditions, the author has not attempted to use the artificial insulating joints in these situations.

In determining the resistance to earth of the cables on each side of the insulating joint, the potentials are assumed to be those that are a result of the sheath current. Since this is seldom 100 percent true, a more accurate measurement of resistance will be obtained by applying sufficient current to make the potential to earth several times greater than the galvanic potentials. By the change in potential to earth and the associated change in current flow each direction from the point of application, the respective resistance to earth can be determined. This method is often used in the measurement of power station grounds.

Since the equipment set-up for creating an artificial insulating joint can be used readily for determining the grounding resistance, the opportunity to obtain a measure of one more factor important in stray current corrosion work should not be overlooked.

DISCUSSION

Question by David T. Jones, Pacific Telephone and Telegraph Co. (Southern Area), Los Angeles, Calif.:

Has the method been used in multiple cable run? Even though all cables are bonded together in each manhole it has been found that the current may be in opposite direction in some cables from the others. In this case it would be necessary that the common bond at the splice be broken and new bonds placed on the cable away from the splice and then an opposing current I_3 be applied which would be the algebraic sum of all the currents in the cable run.

In a cable run having a number of cables, say 10 to 12, this method of inserting artificial insulating joints would be laborious and costly. In our area it has been found to be more practical and advantageous to install insulating joints in all laterals to subscribers' buildings prior to placing the cable run under protection. It also has been found that the remoteness of a ground on the cable and/or cables materially affects the efficacy of the cathode protection.

Reply by G. H. Cantwell:

The method has been used in multiple cable runs. However, the problems you describe were not present. The tests were made on a run located in a residential section of the town where other underground structures had not developed into a closely matted network. Also, the current on the cables was in the same direction and had practically equalized.

I agree with you, that under the conditions you describe, it is more practical to place insulating joints in the laterals to subscribers' buildings.

I wish to point out that I am not attempting to outline the various detail arrangements necessary to make the method work under unusual conditions since these conditions would be many of various degrees and that the engineer with a thorough understanding of the method would recognize the limitations and make the required changes such as you have outlined in your comment.

It is true that the remoteness of a ground on the cables has a direct bearing on the efficacy of the cathodic protection and in these cases the effect of the grounding can be measured by the application of an artificial insulating joint.

Any discussions of this article not published above will appear in the December, 1957 issue.

Corrosion of Austenitic Stainless Steels in Sulfuric Acid*

By E. H. PHELPS and D. C. VREELAND

Introduction

IT HAS BEEN known for years that the stainless steels, particularly the austenitic grades, possess outstanding resistance to the corrosive action of many chemical solutions. In a number of instances, however, the behavior of stainless steels over a wide range of concentrations, temperatures, degrees of aeration, and in the presence of impurities has not been well established.

Sulfuric acid is one of the most widely used inorganic acids. The corrosion resistance of stainless steels in sulfuric acid has been studied by a number of authors, including Kiefer and Renshaw,[1] Fontana,[2] and Tofaute and Rocha.[3] Edeleanu[4] has investigated the potential behavior of stainless steels in sulfuric acid. Of particular interest is his paper[5] on corrosion monitoring of chemical plant equipment in sulfuric acid service. In this method, continuous potential measurements are made on equipment in operation, and are used to indicate the nature of operating conditions.

This paper will present additional data on the corrosion resistance of austenitic stainless steels in sulfuric acid over a wide range of acid concentrations and temperatures, in the "air-saturated" and "air-free" conditions. Preliminary results on the use of potential measurements to determine the "corrosion condition" of austenitic stainless steels in sulfuric acid also will be discussed.

Experimental

Materials

Six types of stainless steel, namely AISI Types 304, 310, 316, 317, 321, and 347, were used in the present investigation. The chemical composition of the materials is listed in Table 1. All these steels were obtained from regular mill products and are representative of commercially available materials.

Reagent-grade sulfuric acid, having a specific gravity of 1.84, and distilled water were used to make all test solutions.

Weight-Loss Tests

The test specimens were sheared from 1/8-inch-thick cold-rolled sheets that had been solution-annealed at 1950 F. One- by two-inch specimens were prepared for testing by successively grinding on 60- to 80-grit and 100- to 120-grit belts. They were then hand-polished with 120-grit emery paper. All specimens were prepared on a dry-belt grinder, except specimens for special tests designed to check the effect of surface preparation; these were prepared on a wet-belt grinder. The specimens were degreased in acetone, cleaned in 20 percent nitric acid at 140 F for 20 minutes, rinsed, dried, and weighed. The nitric acid cleaning was included in the specimen preparation as a passivating treatment, and to obtain uniformly clean surfaces on the specimens.

The corrosion tests were conducted in air-tight glass vessels, each of which contained seven liters of the sulfuric acid solution. Specimens were suspended from glass hooks in each vessel and completely immersed in the solution. A constant temperature was maintained by placing the test vessels in a thermostatically controlled water bath.

The tests were conducted in air-free and in air-saturated solutions. The air-free condition was produced by saturating the solution with purified nitrogen; this served to remove air from the solution. The nitrogen, purified by passing over copper turnings at 450 C, was introduced as fine bubbles into the solution through a sintered-glass diffusion disk. A glass chimney prevented impingement of the bubbles on the specimens. For 48 hours prior

★ Submitted for publication March 20, 1957. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

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D. C. VREELAND—Technologist, Applied Research Laboratory, U. S. Steel Corporation, Monroeville, Pa. He received a BS from Antioch College and an MS in metallurgy from Ohio State University. Prior to joining U. S. Steel, Mr. Vreeland was with Westinghouse Electric Corporation, Electronic Tube Division, and Oak Ridge National Laboratory, where he conducted research on corrosion in liquid metals and fused salts. His present work deals primarily with the corrosion of stainless steel.

Abstract

The corrosion resistance of six different grades of stainless steel was investigated in air-saturated and nitrogen-saturated sulfuric acid solutions at 30, 50, and 70 C. The results for the various grades are presented in terms of "active-passive" regions that depend on acid concentration, temperature, and type of aeration. The use of potential measurements for the determination of the "active-passive" regions, and the use of potential measurements in sulfuric acid plant equipment for detection of severe corrosion conditions are also discussed. 4.3.2

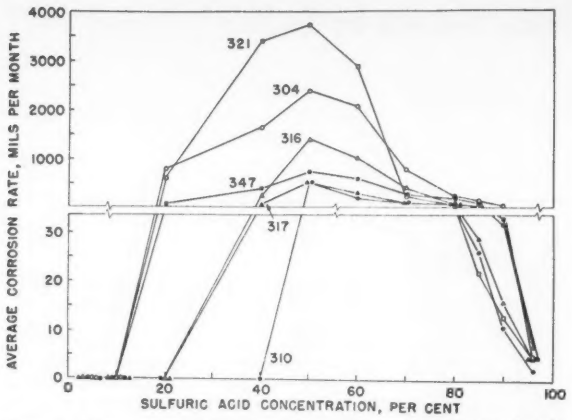

Figure 1—Average corrosion rates of various annealed, passivated stainless steels in air-saturated sulfuric acid at 70 C.

to immersion of the specimens, nitrogen was bubbled through the solution at a rate of 200 ml per minute. This rate of flow of nitrogen was used throughout the test. Although oxygen analyses were not made on solutions treated in the above manner, it is considered that the procedure used substantially removed all oxygen from the test flask.

The air-saturated condition was obtained in a like manner by using air in place of nitrogen at a flow rate of 1200 ml per minute. In some of the tests, carbon dioxide was removed from the air by means of a drying tower packed with Ascarite (granulated sodium hydroxide and asbestos). Because no noticeable variation was observed in corrosion rates with this procedure, most of the tests were made without removing the carbon dioxide.

Six specimens, one of each type of stainless steel, were included in each test. Usually, the tests were conducted for 96 hours, but specimens that corroded very rapidly were removed after 24 hours. All tests were repeated with duplicate specimens.

After being removed from the solutions, the specimens were rinsed, freed of any adherent corrosion products, solvent degreased, and reweighed. The average corrosion rate in mils (0.001 inch) per month was calculated from the loss in weight.

Potential Measurements

Potential measurements were made in the same type of flask as that used in the weight-loss tests. The specimens were about 1 inch by 2 inches with a 10-inch by ⅛-inch stem that was used to make an electrical connection. In all cases, the 1-inch by 2-inch area was kept about 3 inches below the level of the solution. Although it was realized that this method involves a solution-air interface, it was considered preferable to methods involving masking and the resultant crevice effects.

The corrosion potential of the stainless-steel specimens was measured against a saturated calomel electrode immersed in the test solution. A high-impedance vacuum-tube voltmeter was used for all potential measurements.

The potential-measurement experiments were started at room temperature; the temperature was then increased in steps until all specimens were visibly corroding (hydrogen evolution). In most cases, the specimens were activated at each temperature level by touching them briefly with an iron wire. Measurements were made periodically at each temperature level to determine whether the corrosion potential was stable with time. Usually, the temperature was held at a given level for about one day.

Results and Discussion

The corrosion rates of the annealed, passivated, stainless steels in sulfuric acid at 30, 50, and 70 C are shown in Tables 2, 3 and 4. The weight-loss data for the various steels in air-saturated sulfuric acid at 70 C are also shown in Figure 1, which is representative of the behavior at other temperatures. These data show that as the concentration of sulfuric acid is increased, the steels remain passive (negligible corrosion rates) until a critical concentration is reached. For any given alloy, tests at greater concentrations result in excessively high corrosion rates, except at very high acid concentrations. The critical concentration appears to depend on alloy content. For example, in Figure 1, Types 304, 321, and 347 stainless steels corroded at high rates when tested at concentrations above 10 percent; Types 316 and 317 corroded at high rates when tested at concentrations above 20 percent; Type 310 corroded at a high rate when tested at concentrations above 40 percent.

At very high acid concentrations the corrosion rates tended to decrease. This decrease probably is due to decreased ionization of the acid, and possibly to other factors.

Temperature and Aeration

The effect of temperature and aeration with nitrogen or air on the limiting concentration for passivity of Type 317 stainless steel is shown in Figure 2. In this figure, the lines delineate passive regions from active regions, on the basis of the weight-loss results. The metal is considered passive when it has a negligible corrosion rate (less than 0.01 mil per month). It is apparent that temperature and aeration have a marked effect on the limiting concentration for passivity.

The pronounced effect of aeration is contrary to the results of Kiefer and Renshaw,[1] who did not detect appreciable differences in

TABLE 1—Composition of Materials (Percent)

Material No.	Type	C	Mn	P	S	Si	Ni	Cr	Others
D1	304	0.06	0.44	0.022	0.012	0.51	9.0	19.2	.....
D6	310	0.07	1.7	0.022	0.009	0.36	22.0	24.8	.....
D8[1]	316	0.05	1.3	0.010	0.008	0.46	10.7	18.3	2.5 Mo
D1247[2]	316	0.06	2.0	0.017	0.017	0.69	13.4	17.8	2.2 Mo
D04	317	0.06	1.5	0.026	0.013	0.66	13.4	19.0	3.2 Mo
D08	321	0.06	1.3	0.040	0.007	0.44	10.7	17.9	0.54 Ti
D14	347	0.07	1.5	0.018	0.015	0.28	11.7	18.3	0.89 Cb + Ta

[1] Material used in weight-loss tests.
[2] Material used in potential-measurement tests.

TABLE 2—Average Corrosion Rates[1] (Mils per Month) of Annealed, Passivated Stainless Steels in Sulfuric Acid at 70 C

Sulfuric Acid, Percent by Weight	TYPE 304		TYPE 310		TYPE 316		TYPE 317		TYPE 321		TYPE 347	
	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free
0.1	<0.01[2]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
0.5	<0.01	20	<0.01	6.1	<0.01	<0.01	<0.01	<0.01	<0.01	8.5	<0.01	20
1.0	<0.01	23	<0.01	7.0	<0.01	0.71	<0.01	1.2	<0.01	9.2	<0.01	28
5.0	<0.01	55	<0.01	10	<0.01	3.7	<0.01	2.0	<0.01	33	<0.01	44
10.0	<0.01	220	<0.01	68	<0.01	7.0	<0.01	3.1	<0.01	93	<0.01	55
20.0	796	800	<0.01	110	<0.01	14	<0.01	3.9	590	700	100	150
40.0	1650	—	<0.01	—	240	—	76	—	3400	—	410	—
50.0	2400	2900	510	580	1410	1500	490	600	3750	4200	750	850
60.0	2100	—	220	—	1050	—	240	—	2900	—	580	—
70.0	800	—	115	—	410	—	125	—	230	—	290	—
80.0	240	300	36	44	115	130	66	74	48	54	210	230
85.0	120	—	26	—	58	—	29	—	22	—	84	—
90.0	42	—	11	—	32	—	16	—	13	—	33	—
96.0	5.3	5.5	2.3	3.0	7.1	12.6	4.6	8.4	5.0	5.3	4.4	4.9

— No data.
[1] Calculated from losses of weight.
[2] <0.01 = no measurable corrosion.

TABLE 3—Average Corrosion Rates[1] (Mils per Month) of Annealed, Passivated Stainless Steels in Sulfuric Acid at 50 C

Sulfuric Acid, Percent by Weight	TYPE 304		TYPE 310		TYPE 316		TYPE 317		TYPE 321		TYPE 347	
	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free
0.1	<0.01[2]	0.48	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.23	<0.01	0.53
0.5	<0.01	0.90	<0.01	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	0.50	<0.01	0.71
1.0	<0.01	5.0	<0.01	0.07	<0.01	<0.01	<0.01	<0.01	<0.01	1.0	<0.01	0.97
5.0	<0.01	20.5	<0.01	0.42	<0.01	1.54	<0.01	0.51	<0.01	22.8	<0.01	....
10.0	<0.01	33.4	<0.01	1.24	<0.01	2.18	<0.01	0.69	<0.01	32.0	<0.01	12.2
20.0	85.0	206.0	<0.01	29.7	<0.01	4.4	<0.01	1.0	110.0	207.0	16.0	16.6
40.0	400.0	621.0	<0.01	125.0	170.0	181.0	<0.01	2.2	565.0	774.0	78.5	89.6
50.0	520.0	925.0	<0.01	266.0	250.0	507.0	<0.01	54.1	1000.0	1090.0	210.0	241.0
60.0	340.0	400.0	<0.01	48.4	173.0	204.0	<0.01	72.5	310.0	378.0	80.0	88.3
70.0	78.0	84.2	<0.01	26.1	<0.01	66.1	<0.01	45.7	54.8	65.0	25.5	30.5
80.0	30.5	31.1	<0.01	11.9	<0.01	27.9	<0.01	17.3	25.8	30.7	20.0	23.5
85.0	1.14	2.9	<0.01	1.1	<0.01	2.5	<0.01	1.4	1.2	3.1	1.2	1.9
90.0	1.03	1.4	<0.01	0.42	<0.01	1.1	<0.01	0.54	1.1	1.0	0.40	0.65
96.0	<0.01	<0.01	<0.91	<0.01	<0.01	<0.01	<0.01	<0.01	1.1	1.1	<0.01	<0.01

[1] Calculated from losses of weight
[2] <0.01 = no measurable corrosion.

TABLE 4—Average Corrosion Rates[1] (Mils per Month) of Annealed, Passivated Stainless Steels in Sulfuric Acid at 30 C

Sulfuric Acid, Percent by Weight	TYPE 304		TYPE 310		TYPE 316		TYPE 317		TYPE 321		TYPE 347	
	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free	Aerated	Air Free
0.1	<0.01[2]	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
0.5	<0.01	0.73	<0.01	0.05	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.74
1.0	<0.01	1.0	<0.01	0.06	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	1.0
10.0	<0.01	16.7	<0.01	0.80	<0.01	0.15	<0.01	0.07	<0.01	0.80	<0.01	3.3
20.0	<0.01	33.3	<0.01	1.2	<0.01	0.32	<0.01	0.17	<0.01	20.0	<0.01	13.3
40.0	<0.01	46.4	<0.01	4.8	<0.01	14.2	<0.01	0.23	<0.01	31.7	<0.01	23.2
50.0	<0.01	332.0	<0.01	39.6	<0.01	70.5	<0.01	4.5	<0.01	420.0	<0.01	58.3
70.0	<0.01	37.0	<0.01	32.6	<0.01	25.5	<0.01	17.0	<0.01	38.5	<0.01	16.5
80.0	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02
85.0	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02
90.0	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02	<0.01	0.02
96.0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

[1] Calculated from losses of weight.
[2] <0.01 = no measurable corrosion.

corrosion rate when their test solutions were air- or hydrogen-saturated, deaerated, or as-mixed, and consequently ran their tests with acids in the as-mixed condition. It is clear from Figure 2, however, that a difference in result with air- or nitrogen-saturation would not be obtained in a test in which both conditions produce passivity for Type 317 (such as 5 percent sulfuric acid at 30 C). It is also possible that substantially equal corrosion rates would be obtained where nitrogen or air-saturation results in an active condition (such as 25 percent sulfuric acid at 80 C). Data taken from Kiefer and Renshaw's paper also are plotted in Figure 2. The intermediate position of the curve based on their data probably is due to air in the solutions when tested in the as-mixed condition.

Active-passive regions for Types 316 and 304 stainless steels are shown in Figure 3. Results for Types 347 and 321 were similar to that shown for Type 304 in air-saturated acid. These steels did not

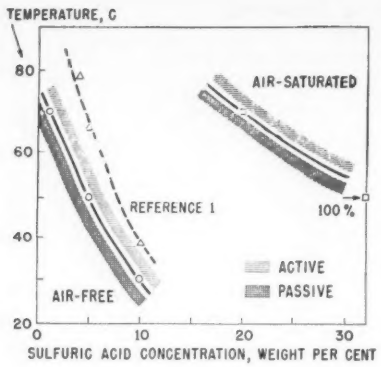

Figure 2—Active-passive regions for Type 317 stainless steel in air-free and air-saturated sulfuric acid.

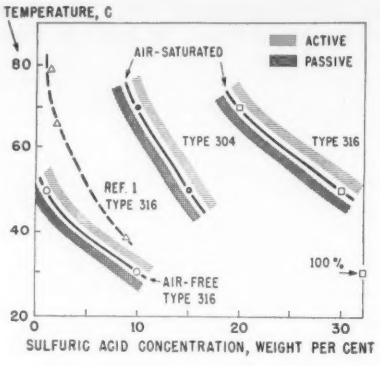

Figure 3—Active-passive regions for Types 304 and 316 stainless steels in air-free and air-saturated sulfuric acid.

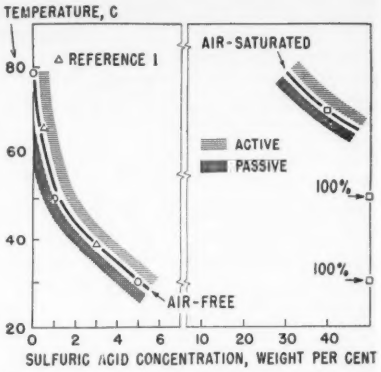

Figure 4—Active-passive regions for Type 310 stainless steel in air-free and air-saturated sulfuric acid.

TABLE 5—Effect of Surface Preparation on Average Corrosion Rates[1] (Mils per Month) of Annealed, Passivated Stainless Steels in Air-Saturated Sulfuric Acid at 70 C

Sulfuric Acid, Percent by Wt.	TYPE 304		TYPE 310		TYPE 316	
	Specimen Prepared on Dry Grinder	Specimen Prepared on Wet Grinder	Specimen Prepared on Dry Grinder	Specimen Prepared on Wet Grinder	Specimen Prepared on Dry Grinder	Specimen Prepared on Wet Grinder
40	1650.0	0.019	<0.01[2]	0.036	240	0.038
50	2400.0	0.041	510	0.017	1410	0.037
60	2100.0	470	220	0.019	1050	0.038

[1] Calculated from losses of weight.
[2] <0.01 = no measurable corrosion.

exhibit an appreciable passive behavior in nitrogen-saturated acid, and hence are not represented for this condition in Figure 3. For Type 316, air saturation markedly extends the passive region. Again, the data of Kiefer and Renshaw for as-mixed acid is intermediate between the data for the air-saturated and the nitrogen-saturated conditions in the present work.

Figure 4 shows the active-passive regions for Type 310 stainless steel. With this steel, the results from the authors' work in the air-free condition and the results from Reference 1 in the as-mixed condition are similar. Comparison of the results for Type 310 with those for Types 316 and 317 in Figures 2 and 3 shows a smaller passive region for Type 310 in the air-free condition than for Types 316 and 317, and a larger passive region for Type 310 in the air-saturated condition than for Types 316 and 317. In other words, the more highly alloyed Type 310 appears more responsive to the effects of oxygen in sulfuric acid than does Type 316 or Type 317.

Surface Preparation and Other Factors

Table 5 shows the results of tests in which specimens prepared with a wet-belt grinder were compared with specimens prepared with a dry-belt grinder. In all but one instance, specimens prepared on a wet-belt grinder had lower corrosion rates than did specimens prepared on a dry-belt grinder. In many instances, the differences obtained was very large. Tarasov[6] states that improper grinding, resulting in high metal-surface temperatures, can cause stresses that accelerate the attack of etchants, and has observed the selective solution of metal in regions that have been highly stressed in grinding.

A number of other factors could have a large effect on the corrosion resistance of stainless steel in sulfuric acid. Among these are variable alloy content in various heats of the same grade of steel, intergranular precipitation due to heat-treatment, surface cleanliness, and crevices if present. It is also known that factors related to the acid, such as purity and the presence of additives, can have an appreciable effect.

Inasmuch as these factors are extremely difficult to control in the commercial use of stainless steels for sulfuric acid service, it is believed that the use of laboratory data, such as those presented in this paper, is questionable in predicting good or poor service behavior. However, general effects shown in laboratory tests, such as those of acid concentration, temperature, type of aeration, and the alloy content of the steel, undoubtedly are important factors in the service behavior of equipment.

Use of Potential Measurements to Determine Service Behavior

The results of weight-loss tests have shown that for the grades of stainless steel tested, a fairly distinct line can be drawn separating active regions (high corrosion rates) from passive regions (negligible corrosion rates). The corrosion rates in active regions are sufficiently high to produce rapid wastage of equipment, whereas the negligible corrosion rates in passive regions would lead to an expectation of indefinite service life.

Edeleanu[5] has pointed out that potential measurements on sulfuric-acid plant equipment offer a means of continuously determining the corrosion condition of the equipment. This method is based on the corrosion-potential and the corrosion-rate behavior of stainless steel in sulfuric acid, represented in Figure 5 (from Reference 5). This figure shows that in the absence of strongly oxidizing conditions, low corrosion rates correspond to a cathodic range of corrosion potentials, and that high corrosion rates correspond to an anodic range of corrosion poten-

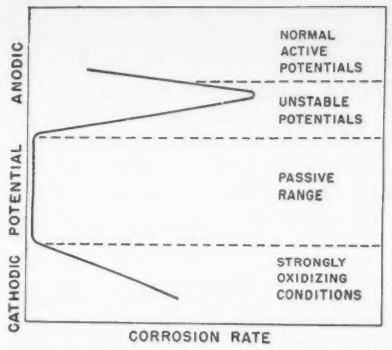

Figure 5—Diagrammatic representation of relation between the corrosion rate and the potential of stainless steel in sulfuric acid (from Reference 5).

tials. Generally, in practice, an anodic corrosion potential would indicate an active corrosion condition in the equipment, whereas a cathodic potential would indicate a passive corrosion condition (again, in the absence of strongly oxidizing conditions). With an anodic-potential value, operating conditions should be changed to produce a less severe corrosion condition.

Because of the obvious advantages of this relatively simple means of corrosion control, it was considered worthwhile to investigate in laboratory tests the potential behavior of a representative stainless steel in sulfuric acid, and to compare this behavior with the weight-loss results previously obtained. For this purpose, Type 316 stainless steel (material number D1247) was used.

Figure 6 is representative of the corrosion-potential behavior of Type 316 in air-saturated 30 percent sulfuric acid. At 25 C and 40 C, the steel maintained a cathodic potential, even after activation. At 50 C, the specimen remained passive for about 20 hours. On activation at this temperature, however, the steel remained active and corroded freely, as indicated by hydrogen evolution.

A different type of behavior was observed at acid concentrations below 30 percent. This is apparent in Figure 7, which shows the behavior of Type 316 in air-saturated 20 percent sulfuric acid. In this experiment, the steel exhibited a cathodic corrosion potential at 30 C and regained a cathodic potential value after activation. At temperatures of 50, 60, and 70 C, the potential remained relatively anodic, but corrosion was not apparent. At 80 C, the potential became somewhat more anodic, and hydrogen evolution was readily visible.

Figure 8 compares the results of the previous weight-loss tests, potential measurements, and points of visible corrosion (as evidenced by hydrogen evolution from specimens in the potential measurement experiments) as a function of temperature and concentration. The active-passive regions indicated by the previous weight-loss tests and by the appearance of visible corrosion in the potential-measurement experiments were similar. The potential measurements, however, indicate a somewhat different separating line at acid concentrations below 30 percent.

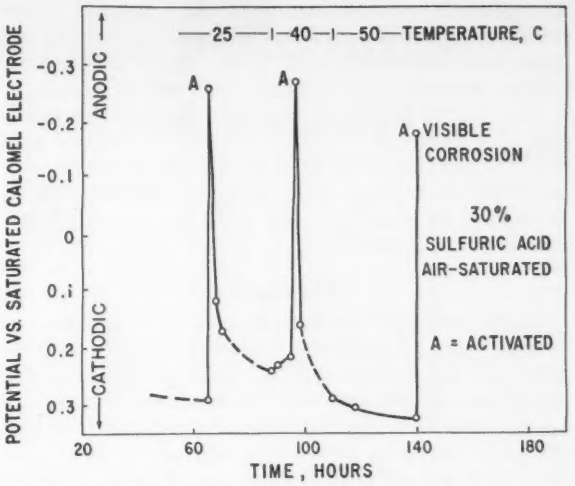

Figure 6—Potential behavior of Type 316 stainless steel in air-saturated 30 percent sulfuric acid at various temperatures.

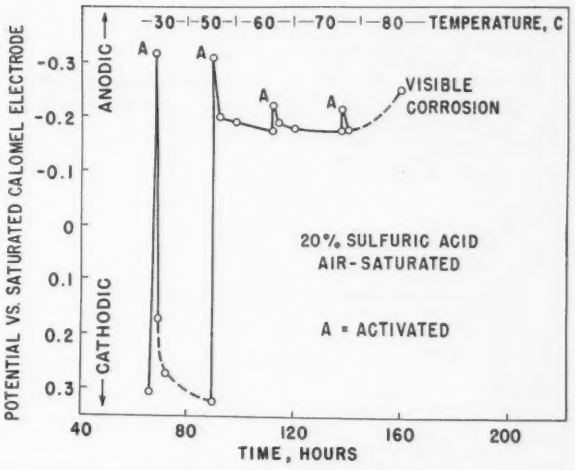

Figure 7—Potential behavior of Type 316 stainless steel in air-saturated 20 percent sulfuric acid at various temperatures.

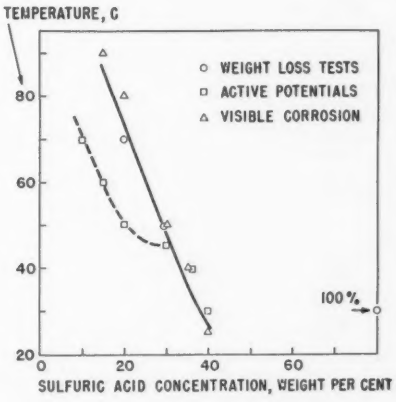

Figure 8—Comparison of potential and corrosion data for Type 316 stainless steel in air-saturated sulfuric acid.

The general correspondence of the potential measurements and the more direct corrosion determinations are considered sufficiently good to warrant further investigation in plant-type service tests.

The use of potential measurements to determine corrosion behavior of stainless steels in sulfuric acid may not apply to certain stainless alloys specifically designed for sulfuric acid service. Further work is necessary to establish the corrosion-potential behavior of these steels.

Summary

It has been shown that the corrosion behavior of certain types of austenitic stainless steels in sulfuric acid can be represented as active-passive regions, dependent on temperature, acid concentration, and type of aeration. For the alloys tested, the extent of the passive region appears to depend on alloy content. Air saturation as compared with nitrogen saturation, significantly extends the range of acid concentration and acid temperature that will produce a passive condition on the alloys tested.

In general, plant operation in which stainless-steel equipment remains passive will result in long service life of the equipment, whereas operation leading to an active condition can be expected to produce severe corrosion damage. Because a large number of factors are involved in predicting the service behavior of stainless-steel plant equipment, the use of potential measurements as a method of continuously determining the corrosion condition was described. Laboratory experiments showed promise for this method.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Standardization in the Field of Corrosion And Corrosion-Protection in Germany*

Abstract

The present status of German standardization relating to the corrosion, corrosion-protection by chemical, electrochemical or galvanic coatings, and testing of metallic industrial materials is tabulated. Some basic information concerning the organization and progress of the work is presented. **1.7.1**

Introduction

DURING THE past few months Task Committee D 5 of the Special Commission for Standardizing Materials Testing (Methods), working under the chairmanship of Professor G. Schikorr, Chemische Landesuntersuchungsanstalt (State Institute for Chemical Research), Kienestrasse 18, Stuttgart, has completed the preparation of a number of standards

TABLE 1—Corrosion in General

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
50,900	Standard	June, 1951	Corrosion of Metals	—
50,901	Draft	May, 1954	Corrosion of Metals, Quantitative Data for Corrosion with Uniform Attack, Concepts and Units	Replaces DIN 4851

TABLE 2—Corrosion Testing Methods

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
50,905	Standard	Nov., 1952	Testing Metallic Industrial Materials, Corrosion Tests, Instructions Concerning Procedure and Evaluation	Replaces DIN 4850
50,907	Standard	Jan., 1952	Testing of Light Metals, Testing of Resistance to Corrosion by Marine Atmospheres and Seawater	Replaces DIN 4853
50,908	Draft	Feb., 1953	Testing of Light Metals, Stress Corrosion Tests	Period for criticism expired on June 30, 1953, not dealt with yet in the Commission
50,909	Standard	Sept., 1954	Testing of Metallic Industrial Materials, Underground Corrosion Tests in the Absence of Stray Currents	Replaces DIN 4860
50,910	Standard	Feb., 1955	Testing of Metallic Industrial Materials, Quantitative Effects and Measuring Techniques for Underground Corrosion in the Presence of Stray Currents	Replaces DIN 4861
4,852	Standard	Aug., 1939	Corrosion, Tests Carried Out in Boiling Liquids (Boiling Test)	New text in preparation
4,854	Standard	Aug., 1942	Corrosion, Testing of Pressure Vessels, Testing at Elevated Pressures and Temperatures	New text in preparation

TABLE 3—Rust Removal, Chemical and Electrochemical Coatings

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
50,940	Standard	Aug., 1952	Corrosion-Protection, Testing of Chemical Rust Removers and Inhibitors (added to mordants or picklers) for Steel and Iron. Laboratory Tests	—
50,942 (with Appendix)	Draft	Oct., 1952	Corrosion-Protection, Instructions Concerning Phosphatizing of Steel Components	Replaces Draft DIN 55,700, rejected as a standard

TABLE 4—Electrodeposited Coatings

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
50,950	Standard	Sept., 1952	Testing of Electrodeposited Coating, Microscopic Determination on Film Thicknesses	—
50,951	Draft	Sept., 1952	Testing of Electrodeposited Coatings, Determination of Film Thicknesses by Radiation Techniques	Rejected as a tentative standard
50,953	Standard	Jan., 1955	Testing of Electrodeposited Coatings, Determination of the Thickness of Chromium Coatings by Means of the Drop Method	—
50,956	Standard	Dec., 1954	Testing of Electrodeposited Coatings, Porosity Testing of Metallized Zinc and Zinc Alloys, Copper Sulphate Process	—
50,960	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Coatings, Symbols, Thickness of Metallic Layers, General Instructions	—
50,960 (appendix)	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Coatings, Electrodeposited Coatings on Steel, General Instructions Concerning their Use in Central and Western Europe as a Protection against Atmospheric Corrosion	—
50,961	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Zinc Coatings on Steel	—
50,962	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Cadmium Coatings on Steel	—

(Table 4 Continued on Page 28)

* Translated from *Metall*, **9**, No. 13/14, 593 (1955) July, by Henry Hives, Publications Division, Aluminium Laboratories Limited, Kingston, Ontario.

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TABLE 4 (Continued)

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
50,963	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Nickel and Nickel-Chromium Coatings on Steel	—
50,964	Tentative standard	Jan., 1955	Corrosion-Protection, Electrodeposited Copper-Nickel and Copper-Nickel-Chromium Coatings on Zinc and Zinc Alloys	
50,970	Draft	Mar., 1953	Electrodeposited Coatings, Nickel Salts for Nickel Baths	Second draft under preparation
50,971	Standard	Oct., 1954	Electrodeposited Coatings, Chemicals for Zinc Cyanide Baths	—

TABLE 5—Metallic Coatings on Wire

German Standard No. (DIN)	Type	Date of Publication	Title	Status of Preparation and Remarks
51,213	Standard	June, 1940	Testing of Wires, Metal Coatings	—

which are either revisions of previous standards concerning corrosion, or new standards relating to the field of electroplating. The latter were published as drafts several years ago. The status of standardization as of February 1955 is tabulated in Tables 1-5.

The standards are available at the Beuth-Vertrieb, Uhlandstrasse 175, Berlin W 15, and Friesenplatz 16, Cologne.

Any discussions of this article not published above will appear in the December, 1957 issue.

Corrosion and Metal Transport In Fused Sodium Hydroxide*

Part 2 – Corrosion of Nickel-Molybdenum-Iron Alloys

By G. PEDRO SMITH* and EUGENE E. HOFFMAN*

Abstract

Studies were made of the corrosion by fused sodium hydroxide of nickel-rich alloys whose constituents were nickel, molybdenum, and iron exposed at 815 C for 100 hours. The reaction mechanism was found to involve the selective removal of iron and molybdenum from solid solution with nickel and a resultant formation of subsurface porosity. In most cases this porosity was shown to occur in the form of elongated pits which frequently followed tortuous paths and intersected to form a maze of subsurface channels.

Alloys of nickel, molybdenum, and iron containing at least 70 percent nickel showed more resistance to corrosion by sodium hydroxide melts at 815 C than any other alloys which have been tested. 4.7

Introduction

IT HAS BEEN known for some time that nickel is relatively corrosion resistant toward fused sodium hydroxide. Recent studies[1] have shown that this corrosion resistance is very good up to high temperatures provided that hydrogen is not removed from the field of reaction and provided that metal transport phenomena are avoided. However, elemental nickel has low mechanical strength at elevated temperatures and cannot be used as a material of construction where excessive thickness is to be avoided. Therefore, it is desirable to impart added strength to the nickel by alloying with other metals. Unfortunately, potential alloying elements are relatively reactive with hydroxide melts even after alloying with nickel.

In this paper it will be shown that certain nickel-rich alloys whose constituents are nickel, molybdenum, and iron show a more promising corrosion resistance at 815 C than alloys which have previously been tested, although elemental molybdenum and iron react quite readily with fused sodium hydroxide at elevated temperatures.[2] It should be emphasized that corrosion rates in fused sodium hydroxide are sensitive to many variables such as the composition of the atmosphere above the melt. Therefore, although the tests to be described show that certain alloys are relatively corrosion resistant, the quantitative data are meaningful only for the test conditions specified.

Experimental Procedure

A study was made of the action of fused sodium hydroxide on the elemental metals nickel and iron and on nine alloy compositions of nickel-rich alloys containing nickel, molybdenum, and iron. The corrosion testing procedure was the capsule test technique described in another report.[3]

The commercial materials A-nickel and Globiron were used for tests on the elemental metals. Alloys were prepared as 30-lb ingots from high-purity materials by vacuum melting and casting. These ingots were extruded into rods, which were machined into capsules and specimens for testing. The nominal compositions in weight percent of the nine alloys tested were as follows:

90 percent Ni-10 percent Mo
85 percent Ni-15 percent Mo
80 percent Ni-20 percent Mo
75 percent Ni-25 percent Mo
80 percent Ni-20 percent Fe
67 percent Ni-33 percent Fe
80 percent Ni-10 percent Mo-10 percent Fe
70 percent Ni-15 percent Mo-15 percent Fe
60 percent Ni-20 percent Mo-20 percent Fe

Five of these alloys were analyzed, and the results are given in Table 1. It will be noted that the actual composition of individual constituents did not deviate by more than 1.52 percent from the nominal composition and that contaminants were present only in small amounts.

Test samples were exposed at 815 C for 100 hours. Duplicate tests were made with each alloy. After the tests the capsules were cut open, the hydroxide was dissolved in water, and the test specimens were weighed. Then the specimens were cut in half. One half was metallographically prepared for microscopic examination; the other half was prepared for X-ray examination as follows: the cut surface was etched with aqua regia to remove strained layers, special precautions being taken to protect the uncut surfaces. A high-angle spectrometer was used for making X-ray examinations of the cut and uncut surfaces. The cut surface was representative of the

★ Submitted for publication April 29, 1957.
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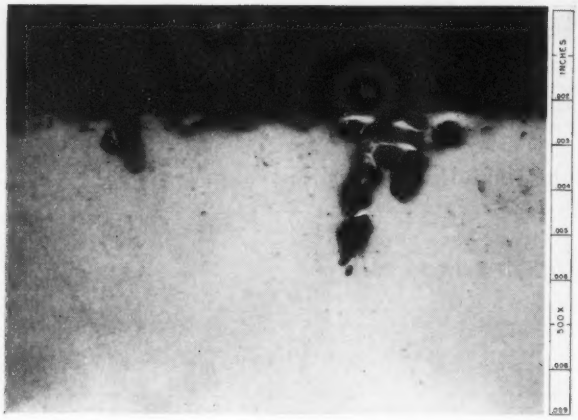

Figure 1—Pits formed on a specimen of 85 percent Ni—15 percent Mo alloy by corrosion in fused NaOH. Specimen is in as-polished condition. Original magnification, 500X; actual magnification after reduction for engraving purposes, 260X; unetched.

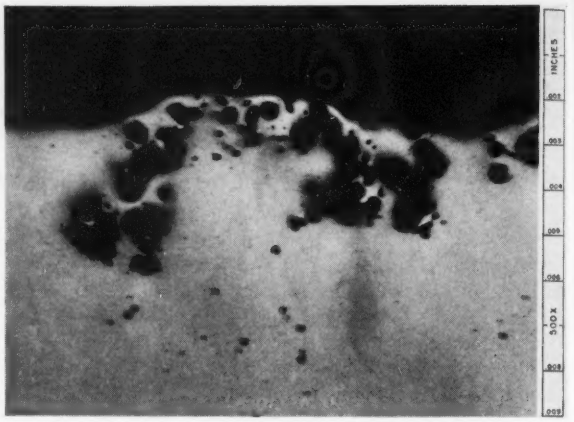

Figure 3—Labyrinthian pits formed on a specimen of 70 percent Ni—15 percent Fe—15 percent Mo by corrosion in fused NaOH. Specimen is in as-polished condition. Original magnification, 500 X; actual magnification after reduction for engraving purposes, 260X; unetched.

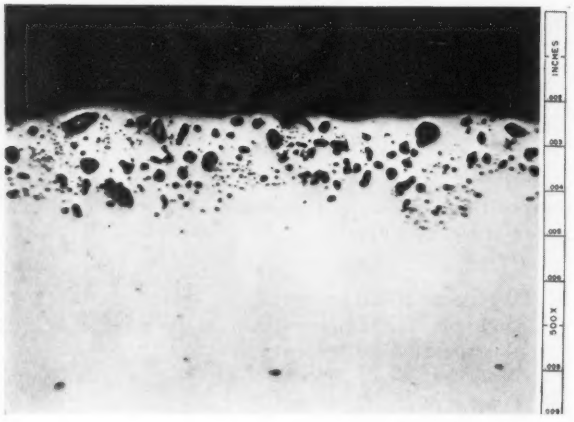

Figure 2—Porous layer formed on a specimen of 75 percent Ni—25 percent Mo alloy by corrosion in fused NaOH. Specimen is in as-polished condition. Original magnification, 500X; actual magnification after reduction for engraving purposes, 260X; unetched.

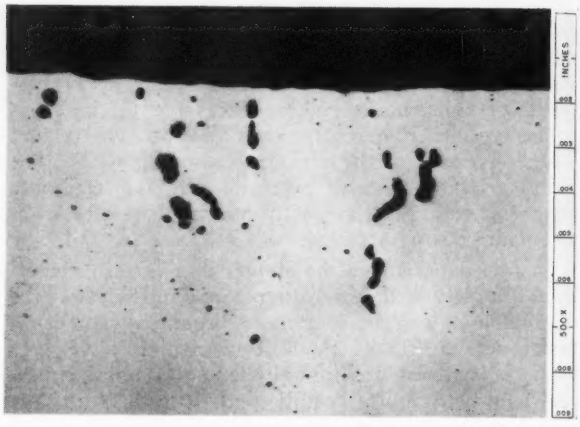

Figure 4—Intergranular porosity formed on a specimen of 60 percent Ni—20 percent Mo—20 percent Fe by corrosion in fused NaOH. Specimen is in as-polished condition. Original magnification, 500X; actual magnification after reduction for engraving purposes, 260X; unetched.

uncorroded interior of the specimen, and the uncut surface was representative of the corroded metal.

Results

After testing, the A-nickel specimens were slightly etched in appearance but were free from visible tarnish films. They were found to have lost 0.005 gm/square inch[2] in weight, which, if uniformly distributed over the surface, would be equivalent to a decrease in thickness of 4.5×10^{-3}-inch. Microscopic examination did not reveal any localized attack such as pitting or grain-boundary penetration. The iron specimens, on the other hand, had corroded significantly, evidenced by a uniform decrease in thickness of 0.005-inch as measured microscopically.

The weight loss suffered by the alloy specimens was slight, being 0.021 gm/square inch for one specimen and less than 0.013 gm/square inch for all the others. There was no correlation between weight loss and alloy composition.

Microscopic examination was made of sections cut perpendicular to a corroded surface of each specimen. The 90 percent Ni-10 percent Mo specimens gave no evidence of corrosion other than a slight waviness of the surface, which was less than 0.6 mil deep. The metallographic sections of specimens of all other compositions contained pores near the surface which, for the most part, were sections through elongated, labyrinthian pits. The density, diameter, and

TABLE 1—Compositions of Alloys Tested

Nominal Composition (Weight Percent)	ANALYSIS (Weight Percent)						
	Ni	Fe	Mo	C	Si	Mn	S
85 Ni-15 Mo	86.07	....	15.09	0.021	0.030	<0.002	<0.0001
90 Ni-10 Mo	89.60	....	10.23	0.014	0.030	<0.002	0.0002
80 Ni-10 Mo-10 Fe	78.48	10.14	9.60	0.010	0.040	<0.002	0.0006
60 Ni-20 Mo-20 Fe	59.44	20.38	19.53	0.018	0.040	<0.002	0.0003
70 Ni-15 Fe-15 Mo	71.29	13.83	14.31	0.013	0.023	<0.017	<0.0001

depth of penetration of these pits differed for different alloy compositions.

The distribution density of pores in nickel-molybdenum alloys increased with increasing molybdenum content. Figure 1 at 500X shows pits which formed in a specimen of 85 percent Ni-15 percent Mo alloy. The group of large pores to the right in the photomicrograph was found to be a section through a single labyrinthian pit which was connected to the surface in two discernible places. This was one of the deepest pits found for specimens of this composition. A second large pit, toward the left in the photomicrograph, was of more nearly average length. In addition to the large pits there were a great number of small slender pits averaging about 0.5 mil in depth, about one-third that of the average depth for the large pits. These slender pits may be seen near the surface of the specimen in Figure 1. The large pits were somewhat widely scattered over the surface and, at places, were as much as 30 mils apart.

At the surfaces of specimens of 75 percent Ni-25 percent Mo alloy there was a layer of fairly densely spaced pores with a more or less well-defined lower boundary as shown at 500X in Figure 2. Microscopic examination left no doubt that the large pores in the upper half of this layer were sections through elongated pits and, it is not improbable that all the pores were part of a system of channels.

The microstructures of the nickel-iron alloys (not illustrated in this report) were in many ways like those shown in Figure 2 except that the penetration was much less regular so that there were a substantial number of small regions at the surface beneath which no pores were found.

Microstructures for two compositions of nickel-molybdenum-iron alloys are shown at 500X in Figures 3 and 4. Specimens of the 70 percent Ni-15 percent Mo-15 percent Fe, shown in Figure 3, contained large pits which were more or less isolated. Specimens of 60 percent Ni-20 percent Mo-20 percent Fe, shown in Figure 4, contained pores which were found, by etching the specimens, to lie along intergranular boundaries. Pores which were close together were shown to be sections through single, very elongated pores, and consequently it is supposed that all pores represented sections through deep pits which more or less followed the course of intergranular boundaries.

Quantitative determinations of the depth of corrosive penetration were made with the aid of a projection microscope. An image of a specimen was projected at an accurately determined magnification of 250X, and measurements were made of the image with calibrated scales. These measurements were accurate to within 0.2 mil. For each alloy composition, 16 to 22 sections of surface each 20 mils long were examined and the maximum depth at which pores were found was determined for each section. These values were averaged and are listed in Table 2, together with the greatest single value for the depth of pore formation.

Nickel-molybdenum-iron alloys over the composition range considered in this report are face-centered cubic, substitutional solid solutions. The X-ray measurements showed that the lattice parameter of metal at the corroded surface had shifted substantially away from the parameter for the uncorroded interior, as illustrated by the data in Table 3 which also includes the parameter for pure nickel. The lattice parameters of iron and molybdenum are not comparable with the data in Table 3 since these elements at room temperature have body-centered cubic lattices. It will be seen from Table 3 that the shift in lattice parameter of the alloys caused by corrosion could be interpreted as an enrichment in nickel content.

TABLE 2—Depth of Porosity

Nominal Composition (Weight Percent)			Depth of Porosity (Inches x 10^3)	
Ni	Mo	Fe	Average	Maximum
90	10	..	..	<0.5
85	15	..	1.6	3.2
80	20	..	1.2	2.2
75	25	..	3.0	6.0
80	..	20	3.3	5.2
67	..	33	2.0	4.0
80	10	10	0.6	1.8
70	15	15	3.7	5.8
60	20	20	4.1	5.0

Discussion

The small weight losses suffered by all specimens represent exceedingly small losses of metal and show that corrosion in the form of an over-all thinning of the specimens was negligible compared with corrosion in the form of subsurface porosity. No further quantitative meaning can be assigned to these weight changes inasmuch as different specimens probably had different amounts of solidified hydroxide retained within the surface layer of pits after washing. Aside from the small weight losses, the only evidence of corrosion was the formation of subsurface porosity.

From the measurements on the depth of subsurface porosity (Table 2) it is concluded that the nickel-rich alloys containing nickel, molybdenum, and iron are significantly more corrosion-resistant toward fused sodium hydroxide at 815 C than are any other alloys previously investigated.[1]

The X-ray measurements indicated that, when the nickel-molybdenum-iron alloys had reacted with fused sodium hydroxide, iron and molybdenum were selectively removed from solid solution with nickel. Corrosion measurements showed that the depth of pore formation had increased with decreasing nickel content. These results for the alloys are in agreement with the finding[1,2] that elemental nickel is much more resistant toward reaction with fused sodium hydroxide than are elemental iron and molybdenum. The resistance toward reaction of ele-

TABLE 3—Lattice Parameter Shift

Alloy (Weight Percent)			Lattice Parameter (Å)	
Ni	Mo	Fe	Interior	Surface
85	15	..	3.5706	3.5286
80	10	10	3.5606	3.5400
60	20	20	3.5788	3.5446
100	..	..	3.5238	3.5238

mental nickel as compared with that of elemental iron, as described in the preceding section, was verified in this study for the experimental conditions used for the alloy reactions.

A characteristic feature of the corrosion described in this paper is the formation of pores near the surfaces of the alloy specimens. In the majority of instances these pores were shown to be sections through labyrinthian pits. In the case of the 60 percent Ni-20 percent Mo-20 percent Fe alloy, illustrated in Figure 4, the pits seemed to follow intergranular boundaries.

The selective removal of reactive constituents from substitutional solid solution is known to lead, frequently, to the formation of subsurface porosity as illustrated by the removal of copper from copper-gold alloys by reaction with aqueous ferric chloride[4,5] and the removal of nickel from iron-chromium-nickel alloys by dissolution in liquid lead.[6] The mechanisms by which subsurface porosity may develop as a consequence of different selective removal processes have not been treated fully, but several important special cases have been considred. One was the formation of voids not connected with the alloy-corrodent interface, as described by Brasunas,[7] but this mechanism does not seem to be operative for the selective removal process under consideration because the evidence is that pits, not voids, were formed.

Another case, described by Bakish and Robertson,[5] was the possible role which galvanic potentials and structural defects play in producing subsurface porosity when the rate of selective removal is controlled by an electrochemical mechanism. Wagner[8] has considered the case in which the rate of selective removal is controlled by the rate of diffusion of the substance being removed, first through the alloy and then through the corrodent phase. He showed that if the diffusivity in the alloy is much less than the diffusivity in the corrodent phase, as is true for liquid corrodents, then corrosion by a pitting mechanism, once initiated, is self-sustaining whether or not there exist inhomogeneities such as structural defects or local concentration differences. Wagner does point out, however, that if there are inhomogeneities present in the alloy they might act as preferred paths for pit penetration.

The above considerations suggest that the penetration of the corrodent into pits in the alloy, such as was observed in the corrosion of nickel-molybdenum-iron alloys by sodium hydroxide, is characteristic of the selective removal of alloy constituents from substitutional solid solution.

Conclusions

Alloys of nickel, molybdenum, and iron containing at least 70 percent nickel showed more resistance to corrosion by sodium hydroxide melts at 815 C than any other alloys which have been tested. The amount of corrosion decreased with increasing nickel content.

The reaction mechanism was found to involve the selective removal of iron and molybdenum from a terminal solid solution with nickel. This process resulted in the formation of a porous alloy at the surface which was much enriched with nickel. In most cases the porosity was shown to occur in the form of elongated pits which frequently followed tortuous paths and intersected to form a maze of subsurface channels.

Acknowledgment

The authors are indebted to H. Inouye for making the special alloys, to R. J. Gray and N. M. Atchley for preparing the metallographic specimens and taking the photomicrographs, and to H. L. Yakel and R. M. Steel for doing the X-ray work.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Sulfide Corrosion Cracking Of High Strength Bolting Material*

By DONALD WARREN and G. W. BECKMAN

Introduction

THE SPONTANEOUS cracking of relatively high-strength ferrous materials in hydrogen sulfide environments has been termed sulfide corrosion cracking.[1] Such cracking results from the combined action of tensile stresses and corrosive attack by hydrogen sulfide.[1,2]

Failure by sulfide corrosion cracking has been most commonly encountered in the handling of sour crudes in the petroleum industry. Field failures of such equipment as tubing, sucker rods, valve springs and plates, pump springs, bolting, and wire pipe filters have been reported.[2,3,4,5] Not all failures due to hydrogen sulfide have occurred within the petroleum industry, however. For example, the rupture of low alloy steel cylinders charged with coal gas containing about 0.01 percent H_2S has been attributed to sulfide corrosion cracking.[5,6]

The current investigation stemmed from numerous failures of AISI Type 4140 bolting material in aqueous hydrogen sulfide service. Design information was needed to prevent the recurrence of such failures. This information was developed by studying the incidence of cracking in a number of H_2S-H_2O environments. Those variables evaluated were the bolting material, hardness, applied stress, and plastic prestrain. Preventive measures such as the use of metallic coatings, corrosion inhibitors, and substitute materials also were evaluated.

Experimental Details

Test Specimens and Bolting Materials

Bolts were used as test specimens in this study because their stress patterns are difficult to simulate with other types of laboratory specimens. Two types of bolting materials were tested: (1) AISI Type 4140 steel, the material which failed in service, and (2) substitute materials, most of which were nonferrous alloys.

AISI Type 4140 Steel. Allen head cap screws ⅜ inch in diameter by 1½ inches long with 6 threads per inch were used as test specimens. This bolt size was chosen because: (1) similar bolts had failed in the field, and (2) the large number of required test specimens limited the bolt size from the standpoint of economy, ease of procurement, and test space.

All cap screws had been commercially fabricated from a single heat of steel. The chemical composition of the steel is given in Table 1. The hardness of the cap screws in the commercially annealed condition varied from R_c 10 to R_c 20. The cap screws were hardened and tempered to provide bolts with hardnesses in the range of R_c 20 to R_c 55. The yield strength and ultimate tensile strength of the bolts is shown in Figure 1 as a function of hardness and tem-

★ Submitted for publication January 25, 1957. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

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G. W. BECKMAN is a metallurgist with the Atomic Energy Division, Explosives Department, Savannah River Plant, E. I. du Pont de Nemours & Company (Inc.), Augusta, Georgia. He received a BS degree from the University of Massachusetts in 1939 and an MS in metallurgical engineering from Lehigh University in 1949. Mr. Beckman was employed by Chapman Valve Manufacturing Company, Indian Orchard, Mass., from 1940 to 1943 and served as Aerological Engineer in the U. S. Navy from 1943 to 1946. From 1949 to 1950 he was with Karl Lieberknecht Company, Reading, Pennsylvania.

Abstract

The sulfide corrosion cracking behavior of Type 4140 bolts was studied in H_2S-H_2O systems at temperatures of 20 to 120 C and at pressures of 1 to 17 atmospheres. The susceptibility to cracking increased with increasing bolt hardness, increasing applied stress, increasing amounts of plastic deformation, and decreasing test temperature. Other bolting materials such as Inconel, Inconel-X, K-Monel, and annealed Type 316 stainless steel were completely resistant to sulfide corrosion cracking.

The use of protective metallic coatings and the addition of corrosion inhibitors to the test system were evaluated as means of preventing the sulfide corrosion cracking of Type 4140 bolts.

The sulfide corrosion cracking of Type 4140 bolts is believed to be best explained on the basis of hydrogen embrittlement. **3.2.2**

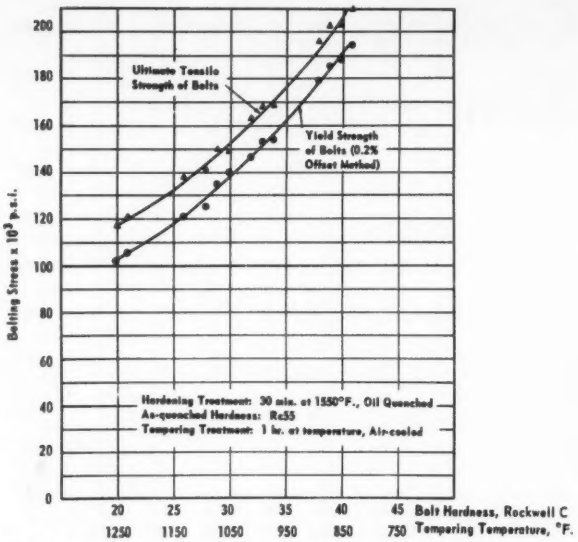

Ultimate Tensile Strength of Bolts
Yield Strength of Bolts (0.2% Offset Method)
Bolting Stress x 10³ p.s.i.
Hardening Treatment: 30 min. at 1550°F., Oil Quenched
As-quenched Hardness: Rc55
Tempering Treatment: 1 hr. at temperature, Air-cooled
Bolt Hardness, Rockwell C
Tempering Temperature, °F.

Figure 1—Physical properties of hardened and tempered Type 4140 bolts.

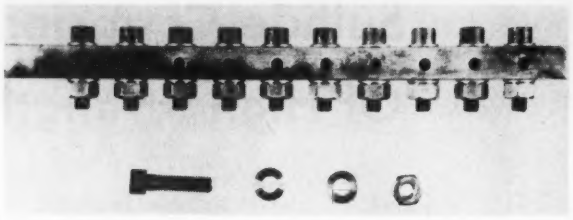


Figure 2—Bar type jig used for loading 3/8-inch Allen head cap screws.

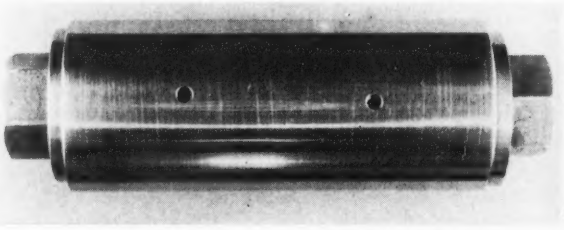


Figure 3—Sleeve type jig used for loading stud bolts.

pering temperature. The strength values were obtained from tensile tests made on the bolts.

Substitute Bolting Materials. The substitute materials evaluated were Type 316 stainless steel, Inconel, Inconel X, K-Monel, 5 percent aluminum bronze, and 75ST aluminum. These materials were tested as ⅜ inch or ½ inch in diameter studs. Their physical properties are included in the discussion of results.

Method of Stressing Bolts

Loading Jigs. Two types of jigs were used for loading (applying stress to) the bolts. The cross-sectional area of both jigs was large enough to provide a rigidity greater than that of the bolts being tested. The ⅜ inch Allen head cap screws were loaded on bars as shown in Figure 2. The longer stud bolts were loaded in individual sleeves as shown in Figure 3. Holes were drilled in both jigs to permit free circulation of the corrosive environment around the bolts.

Misalignment of the bolts during loading on the test jigs was minimized by: (1) machining parallel flats on the surfaces of all bars, sleeves, and washers (2) machining all nut faces to align them perpendicularly to the thread axis; and (3) rechasing all threaded surfaces prior to engagement. To reduce friction, powdered molybdenum disulfide was brushed over all of the mating surfaces.

Technique of Stressing Bolts. Known values of stress were applied to the bolts by tightening them on the jigs until their elongations reached certain predetermined values. The amount of bolt elongation required to give the desired stress was calculated from the following formula derived from Hooke's Law:

$$e = \frac{SL}{E}$$

where e is the bolt elongation, S is the desired stress, L is the effective gage length of the bolt, and E is the modulus of elasticity of the bolting material. The torque required to produce the desired stress was measured by means of a calibrated torque wrench. The torque values were used in checking for bolt failures (to be discussed later).

The elongation of the longer stud bolts was measured directly with a micrometer. The accuracy of these measurements was improved by machining hemispherical tips on the end of each stud bolt. The gage length, L, was assumed to be the length of the loading sleeve plus the thickness of the two washers plus half the total thickness of the two nuts. The use of only half the total nut thickness to determine the effective gage length is based on the work of Stoeckly and Macke[7] and Hetenyi.[8] Their results showed that 70-75 percent of the bolt load was carried by the first half of the threads engaged in the nut.

Special techniques were required to measure the elongation of the ⅜ inch Allen head cap screws under applied loads. A Graham-Mintel strain indicator, which operates on the microformer principle, permitted elongation measurements having an estimated precision of 0.00005 inch. A complete derivation of the formula used for calculating the stresses in the ⅜ inch cap screws is given in the appendix.

Check on Bolt Stressing. The accuracy of the method, used for determining bolting stress was checked by means of the device shown in Figure 4. This device consisted of a cubical block with resistance type strain gages mounted on the four side faces in a direction parallel to the axis of the hole in the block. The block was calibrated by loading it in compression between the platens of a hydraulic testing machine using the adapters shown in Figure 4. The strain on the block as indicated by the strain gages was then measured as a function of the load on the testing machine.

The next step was to load the calibrated block by tightening a ⅜ inch cap screw in place through the

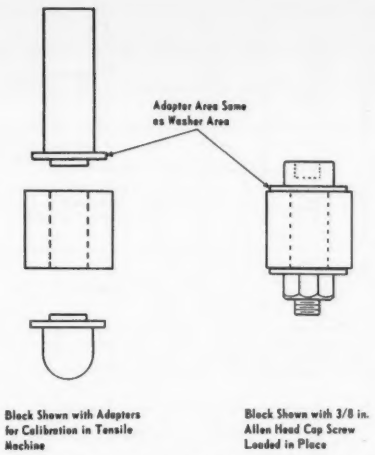

Adaptor Area Same as Washer Area
Block Shown with Adapters for Calibration in Tensile Machine
Block Shown with 3/8 in. Allen Head Cap Screw Loaded in Place

Figure 4—Sketch of calibration block used for checking the strain measurement techniques applied to ⅜-inch Allen head cap screws.

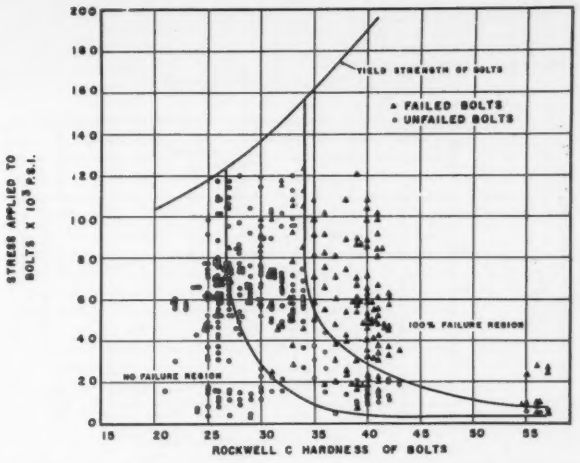

YIELD STRENGTH OF BOLTS
▲ FAILED BOLTS
○ UNFAILED BOLTS
100% FAILURE REGION
NO FAILURE REGION
STRESS APPLIED TO BOLTS X 10³ P.S.I.
ROCKWELL C HARDNESS OF BOLTS

Figure 5—Cracking susceptibility of Type 4140 bolts exposed in an H_2S-H_2O system for one year at 40 C and 250 psi.

hole in the block. As the bolt was tightened, its elongation was measured with the Graham-Mintel strain indicator. At the same time, the strain in the block was determined by means of the resistance strain gages. The bolting stress was then calculated both from the elongation in the bolt and from the load on the block, as provided by the calibration curve. The difference between the values of bolting stress obtained by the two methods of measurement averaged about 7 percent. It was concluded that the strain measurement method of determining bolting stress was sufficiently accurate for the purpose of this study.

Method of Prestraining Bolts

For the prestrain studies, the ⅜ inch cap screws were plastically prestrained in tension in a hydraulic testing machine. As each bolt was prestrained, its stress-strain curve was automatically recorded using a separable extensometer operating over a two inch gage length. The prestraining operation was stopped when the total deformation reached a predetermined amount. The total plastic deformation from the stress-strain curve was then translated into plastic strain in inches/inch for the ¾ inch gage length actually being prestrained.

The amount of plastic prestrain also was determined from reduction in area measurements made with a micrometer and from measurements of the extension between the threads with an eyepiece comparator. All three methods of measuring plastic prestrain yielded approximately the same results until the prestress values approached the ultimate tensile strength of the bolts. The values of plastic prestrain reported here were all based on stress-strain diagrams.

Method of Measuring Bolt Hardness

Earlier studies[1, 2, 9] have emphasized that material hardness is an important variable in sulfide corrosion cracking. Accurate hardness measurements were obtained in this study by machining parallel flats on those surfaces involved in making the hardness determinations. Hardness measurements were made on

TABLE 1—Chemical Composition of ⅜-Inch Allen Head Cap Screws

ELEMENT	Percent By Weight
C	0.41
Cr	0.87
Mo	0.12
Mn	0.80
Si	0.20
S	0.06
P	...

the ends of all bolts prior to testing. In addition, check measurements were made on failed bolts in the area adjacent to the failure.

Test Environments

Previous work[1, 2, 5, 10] has shown that hydrogen sulfide and water are the essential components of systems producing sulfide corrosion cracking. Three principal test environments based on H_2S-H_2O systems were used in the current study. Two of these were pressurized (250 psi) environments operating at temperatures of 40 C and 120 C, respectively. The third system operated at atmospheric pressure and room temperature. The pressurized tests were made in carbon steel autoclaves, while the atmospheric pressure tests were run in sealed steel containers. Commercial grade H_2S was used for all tests.

Preparation of the pressurized systems consisted of purging the test autoclaves with H_2S, then pressurizing them to 250 psi with H_2S, and finally sealing off the systems at that pressure. In the 120 C environment the bolts were exposed to H_2S only when the temperature was 120 C; the system was charged with nitrogen during the heating and cooling cycles.

The room temperature—atmospheric pressure environment was obtained by purging the test container with H_2S and then sealing it off at atmospheric pressure. The concentration of H_2S was maintained by repurging the container with H_2S at periodic intervals.

In the 40 C—250 psi and in the room temperature —atmospheric pressure systems, the bolts were exposed in the vapor rather than the liquid phase.

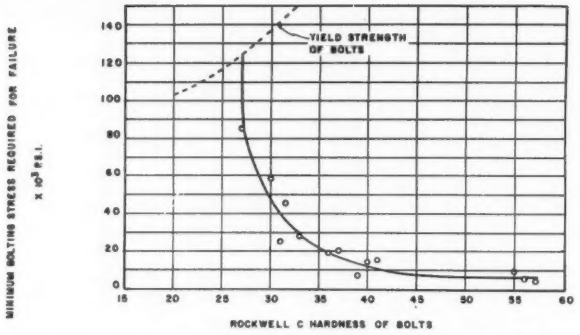


Figure 6—Minimum applied stress required to produce cracking of Type 4140 bolts in an H_2S-H_2O system at 40 C and 250 psi.

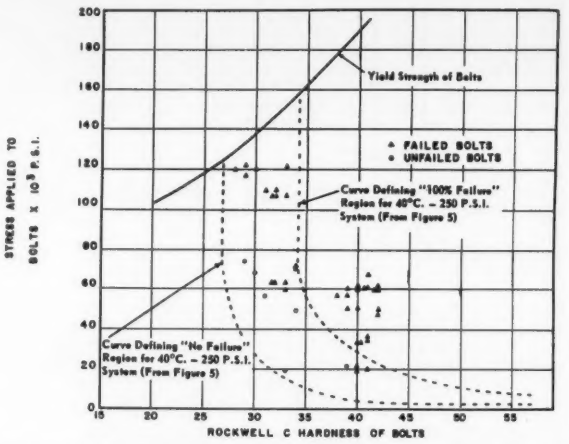

STRESS APPLIED TO BOLTS X 10³ P.S.I.
Yield Strength of Bolts
▲ FAILED BOLTS
○ UNFAILED BOLTS
Curve Defining "100% Failure" Region for 40°C. – 250 P.S.I. System (From Figure 5)
Curve Defining "No Failure" Region for 40°C. – 250 P.S.I. System (From Figure 5)
ROCKWELL C HARDNESS OF BOLTS

Figure 7—Cracking susceptibility of Type 4140 bolts exposed for one year in an H_2S-H_2O system at room temperature and atmospheric pressure

Earlier research[5,6,10] indicated that the vapor phase must be saturated with water if sulfide corrosion cracking is to occur. Saturation of the current test systems with water vapor was ensured by placing a container of water in each of the test pots. In the 120 C—250 psi system, bolts were exposed in both the vapor and liquid phases, as well as a combination of the two. As far as could be determined, there was no difference in the tendency of the vapor or the liquid phases to cause sulfide corrosion cracking at 120 C.

Length of Test Period

The frequency of examination for bolt failures varied from one day in early stages of testing to three months in the latter stages of testing. In most cases, the bolts were exposed for a minimum of 3,000 hours before discontinuing the test.

Method of Checking for Failures

The bolts were checked for failures by torquing each bolt to 75 percent of the torque originally used in stressing it. Occasionally, this procedure failed to detect broken bolts, because the bolts had been locked in place by a heavy layer of sulfide scale. Consequently, when the test was completed, all unfailed bolts were torqued beyond their yield points to reveal any undetected failures (either partial or complete).

Discussion of Results

Cracking Behavior of Type 4140 Bolting Material

Effect of Hardness and Applied Stress. The effect of hardness and applied stress on the sulfide corrosion cracking behavior of Type 4140 bolting material is discussed below for each of the three test environments. Unless otherwise indicated, all data are for quenched and drawn bolts having a microstructure of tempered martensite.

The stress values used are those applied to the bolts before testing. Stress relaxation studies showed that Type 4140 bolts stressed to values below their yield strength underwent a stress relaxation of the order of 3 percent at room temperature and 6 percent at 120 C. About 2-3 percent relaxation occurred during the initial stressing of the bolts at room temperature. The greater relaxation at 120 C was probably due to the lower yield strength of the bolting material at that temperature.[11]

Bolts Exposed at 40 C and 250 psi. Figure 5 shows the cracking susceptibility of Type 4140 bolts in a pressurized (250 psi) H_2S-H_2O system at 40 C. Here, the test bolts are shown as either failing or not failing after a minimum exposure of one year. Two curves have been drawn to divide the plot into three regions: (1) a 100 percent failure region, (2) a borderline region of possible failure, and (3) a no-failure region. Tests were confined to that area below the yield strength curve for the bolts in order to minimize the amount of plastic deformation introduced during stressing.(1) Excessive amounts of plastic deformation would have obscured the effects of hardness and applied stress by accelerating the sulfide corrosion cracking process.[12]

The curves of Figure 5 show clearly that increasing the bolt hardness increased the susceptibility to sulfide corrosion cracking. No bolting failures occurred at hardness values less than R_c 27 even at applied stresses approaching the yield point. This means that in sulfide corrosion service higher bolt loads can be maintained without failure by lowering the bolt hardness to below R_c 27. This contrasts with the usual case where, in the absence of any embrittling phenomena, higher clamping forces are obtained by increasing the bolt hardness and strength level.

The above observations substantiate the work of other investigators showing that steels of higher hardness have a greater tendency toward sulfide corrosion cracking.[1,9,12] In addition, the minimum hardness of R_c 27 for sulfide corrosion cracking of Type 4140 steel is in close agreement with the minimum

(1) Some localized yielding can occur at the thread roots even at applied stresses which are nominally well below the yield point. This is due to the stress concentration effect introduced by the thread root.

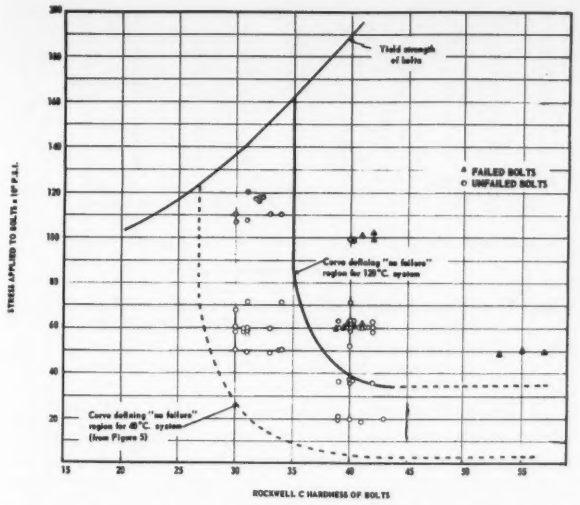

Yield strength of bolts
FAILED BOLTS
UNFAILED BOLTS
Curve defining "no failure" region for 120°C. system
Curve defining "no failure" region for 40°C. system (from Figure 5)
STRESS APPLIED TO BOLTS x 10³ P.S.I.
ROCKWELL C HARDNESS OF BOLTS

Figure 8—Cracking susceptibility of Type 4140 bolts exposed for one year in an H_2S-H_2O system at 120 C and 250 psi.

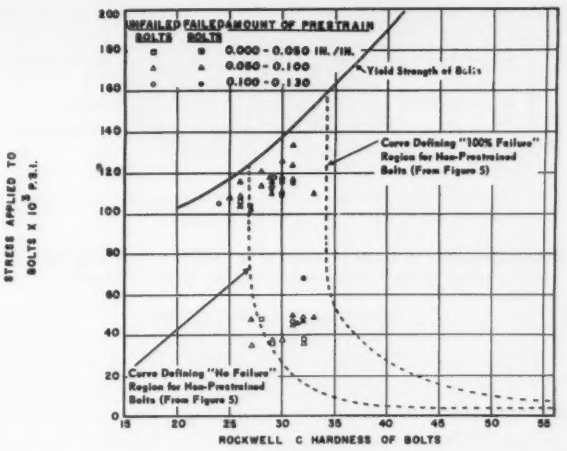

UNFAILED BOLTS
FAILED BOLTS
AMOUNT OF PRESTRAIN
0.000 - 0.050 IN./IN.
0.050 - 0.100
0.100 - 0.130
Yield Strength of Bolts
Curve Defining "100% Failure" Region for Non-Prestrained Bolts (From Figure 5)
Curve Defining "No Failure" Region for Non-Prestrained Bolts (From Figure 5)
STRESS APPLIED TO BOLTS X 10³ P.S.I.
ROCKWELL C HARDNESS OF BOLTS

Figure 9—Effect of prestrain on cracking susceptibility of Type 4140 bolts exposed in an H_2S-H_2O system at 40 C and 250 psi.

value of R_c 24 for cracking of hardenable grades of stainless steel reported by Bloom.[9] Bloom[9] also ran a limited number of tests on Type 4130 steel in a solution of ½ percent acetic acid and H_2S. These tests showed cracking of specimens at hardnesses of R_c 27-28 but not at R_c 21.

From Figure 5 it is evident that the level of applied stress required to produce sulfide corrosion cracking decreased as the hardness increased. Figure 6 illustrates more strikingly this relationship between the bolt hardness and the applied stress necessary for failure. The curve in Figure 6 was obtained by plotting the minimum bolting stress required for failure, as determined from Figure 5, as a function of bolt hardness. Bowers, McGuire, and Wiehe[12] have suggested that there is a "critical" or threshold" stress below which sulfide corrosion cracking will not occur. The data in Figure 6 support their suggestion and show that this "threshold" stress is, in large measure, a function of hardness (or strength level) of the material.

In addition to the above tests on quenched and tempered bolts, more than one hundred annealed bolts were tested for a period of one year without any failures. The annealed bolts had a pearlitic-ferritic microstructure and hardnesses of R_c 15-21. Applied stress values for these bolts ranged from 45,000-80,000 psi (55-95 percent of their yield strength[(2)]).

Bolts Exposed at Room Temperature and Atmospheric Pressure. Figure 7 gives the cracking data for Type 4140 bolts exposed in an H_2S-H_2O system at room temperature and atmospheric pressure. Included in Figure 7 for comparison are the cracking curves from Figure 5 for the 40 C—250 psi environment.[(3)] A comparison of the curves and the plotted data shows very little difference in the cracking behavior of Type 4140 steel in the two environments. Thus, it can be concluded that pressure in the range of 1 to 17 atmospheres has little effect on the cracking tendency. Others[1,2,12] have drawn similar conclusions regarding the effect of pressure over a much broader range (1-300 atmospheres).

TABLE 2—Effect of Plastic Prestrain on the Cracking Susceptibility Of Type 4140 Bolts Exposed in an H_2S-H_2O System at 40 C and 250 Psi

Degree of Prestressing	Amount of Prestrain Introduced, (in./in.)	Range of Applied Stress: 35,000-70,000 psi — Number of Bolts Tested	35,000-70,000 psi — Percent Failed	100,000 psi-Yield Strength — Number of Bolts Tested	100,000 psi-Yield Strength — Percent Failed
None	0	60	8	28	21
Below Ultimate Tensile Strength	0.010-0.030	4	0	4	50
At or Near Ultimate Tensile Strength	0.050-0.070	6	33	11	64
Beyond Ultimate Tensile Strength	0.070-0.130	7	14	11	100

Note: Range of hardness for nonprestrained bolts was R_c 27-33 and for prestrained bolts was R_c 24-33. All bolts were quenched and drawn and had a tempered martensitic microstructure.

Bolts Exposed at 120 C and 250 psi. At 120 C the Type 4140 bolts were far less susceptible to sulfide corrosion cracking than they were at 40 C. This is shown by the cracking data in Figure 8 for bolts exposed in a pressurized (250 psi) H_2S-H_2O system at 120 C.[(4)] Compare the curve defining the no-failure region for the 120 C system with that defining the same region for the 40 C system (the latter curve was taken from Figure 5). It is obvious from these two curves that the cracking susceptibility was greatly reduced when the test temperature was raised from 40 to 120 C.

(2) For a given hardness, the yield strength of annealed bolts was appreciably lower than the yield strength of quenched and drawn bolts.

(3) The data for the room temperature-atmospheric pressure system were believed insufficient for drawing new cracking curves for this system.

(4) The applied stress values used to plot the data in Figure 8 are those measured at room temperature. Relaxation measurements showed that these stress values were reduced by about 6 percent by the 120 C exposure.

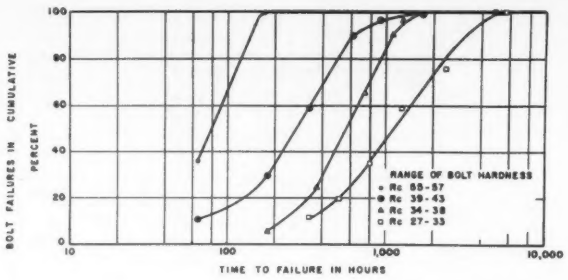


Figure 10—Effect of hardness on time to failure of Type 4140 bolts exposed in an H_2S-H_2O system at 40 C and 250 psi.

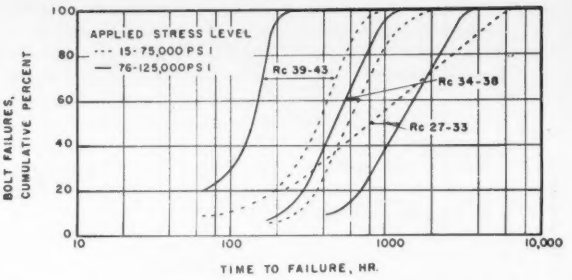


Figure 11—Effect of applied stress on time to failure of Type 4140 bolts exposed in an H_2S-H_2O system at 40 C and 250 psi

Fraser and Treseder[1] also found that the severity of sulfide corrosion cracking decreased with increasing temperature. For example, a 5 percent Ni steel which failed at 0 C in an H_2S-H_2O system did not fail in a 20-25 C system, all other conditions of the exposure being the same.

Effect of Plastic Deformation on Bolts Exposed at 40 C and 250 psi

Heavy plastic prestraining of Type 4140 bolts increased their susceptibility to sulfide corrosion cracking at high levels of applied stress. Figure 9 gives the cracking data for a series of bolts of R_c 24-33 hardness that were plastically prestrained in tension from 0.010 to 0.130 inch/inch and then exposed to a pressurized (250 psi) H_2S-H_2O system at 40 C. Also included in Figure 9 are the cracking curves from Figure 5 for nonprestrained bolts exposed to the same environment. Note that in the absence of plastic prestrain, sulfide cracking did not occur if the bolt hardness was less than R_c 27. However, if bolts were severely prestrained plastically (0.050 inch/inch or more) and then stressed at 100,000 psi or more, sulfide cracking occurred at hardnesses as low as R_c 24. This change in minimum bolt hardness for cracking is too small to be of practical significance. Nevertheless, it does show that plastic deformation and the accompanying residual stresses increase the susceptibility to sulfide corrosion cracking.

In addition to lowering the minimum hardness for failure, plastic prestraining increased the percentage of failures among R_c 24-33 bolts stressed to 100,000 psi and above. This is shown by Table 2 where the bolts have been arranged in three groups based on the amount of prestressing (or prestraining) they received. At applied stress levels of 100,000 psi or more, the percentage of bolt failures increased as the amount of prior plastic deformation increased. In fact, 100 percent of those bolts prestressed beyond their ultimate tensile strength[5] failed when subjected to applied stresses of 100,000 psi. At stress levels of 35,000-70,000 psi plastic prestraining showed no consistent effect on the number of bolt failures. Bowers, McGuire, and Wiehe[12] also found that the effect of prior plastic deformation on sulfide corrosion cracking was highly dependent upon the level of applied stress.

[5] The amount of plastic prestrain introduced was greater than the strain corresponding to the ultimate tensile strength of the bolts.

A limited number of tests involving plastic prestrain were run on annealed bolts having a pearlitic-ferritic microstructure. These tests showed that extreme plastic prestraining (0.180 inch/inch) could cause failure of annealed bolts at hardnesses as low as R_c 22.

The above results emphasize that over-torquing (torquing beyond the yield strength) of bolts should be avoided in field service involving H_2S.

Factors Influencing the Time to Failure of Type 4140 Bolts

The time for failure of Type 4140 bolts in a pressurized H_2S-H_2O system at 40 C ranged from 65 hours to over nine months. Analysis of the cracking data for this test system showed that the time to failure was primarily influenced by bolt hardness and secondarily by stress level and degree of prestressing.

Effect of Hardness on Time to Failure. The curves in Figure 10 illustrate the marked reduction in cracking time caused by increased bolt hardness. These curves were obtained as follows: for each hardness level, the number of bolt failures occurring within a given exposure time was plotted as a percentage of the total number of bolt failures in that hardness range. These data represent bolt failures which occurred at applied stress values ranging from 15,000 to 125,000 psi. A separate analysis of the data showed that the effect of hardness on the time of failure largely overshadowed any effect of applied stress. This is discussed below.

The shorter failure times for the harder bolts probably reflects the greater ease with which cracks can initiate and propagate in a less ductile material.

Effect of Stress Level on Time to Failure. The level of applied stress (below the yield strength) had little effect on the cracking time except for bolts of higher hardness. As shown in Figure 11, an increase in applied stress reduced the time to failure of the R_c 39-43 bolts but did not appreciably affect the time to failure of bolts in the two lower hardness ranges. The latter results are in agreement with those of Gifkins and Rees.[6] They found a poor correlation between the applied stress and time to actual fracture but good agreement between applied stress and the time to initiate sulfide corrosion cracking.

The lack of correlation between the applied stress and time to failure for the softer bolts probably results from the relaxation in bolt load caused by

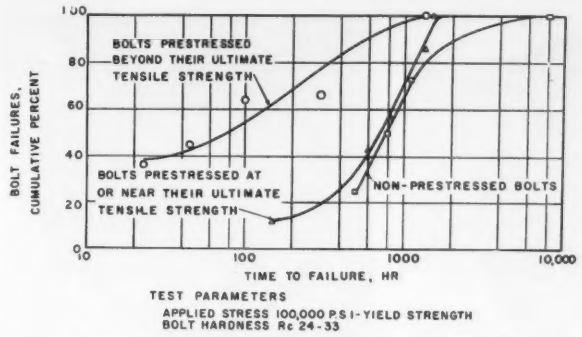

BOLTS PRESTRESSED BEYOND THEIR ULTIMATE TENSILE STRENGTH
BOLTS PRESTRESSED AT OR NEAR THEIR ULTIMATE TENSILE STRENGTH
NON-PRESTRESSED BOLTS
BOLT FAILURES, CUMULATIVE PERCENT
TIME TO FAILURE, HR
TEST PARAMETERS
APPLIED STRESS 100,000 P.S.I - YIELD STRENGTH
BOLT HARDNESS Rc 24-33

Figure 12—Effect of prestressing on time to failure of Type 4140 bolts exposed in an H_2S-H_2O system at 40 C and 250 psi.

TABLE 3—Corrosion Rates for Type 4140 Steel in Three H_2S-H_2O Systems

H_2S-H_2O Environment	Average Corrosion Rate, mil/yr*			
	Short Time Exposure (100-400 hour)		Long Time Exposure (800-1800 hour)	
	Vapor Phase	Liquid Phase	Vapor Phase	Liquid Phase
Room temperature and atmospheric pressure	3	8	2	6
40C and 250 psi	3	9	2	5
120 C and 250 psi	**	15	**	2

* These corrosion rates are the average of three or more tests for the period of time indicated. The test specimens were cylinders ¾ inch in diameter by ¾ inch long. The sulfide scale on the specimens was removed at the end of test period by pickling in an inhibited hydrochloric acid solution.

** Test arrangements in the 120 C-250 psi autoclave did not permit corrosion testing in the vapor phase.

propagation of cracks. As a crack propagates, the unit stress on the remaining cross section of the bolt increases until the yield point of the material is exceeded. The higher yield strength of the harder bolts limits the relaxation of the bolt load and thereby emphasizes the effect of the applied stress.

Effect of Prestressing on Time to Failure. Prestressing of the bolts had little effect on their cracking time unless the prestressing was carried beyond the ultimate tensile strength. Figure 12 shows that prestressing to beyond the ultimate tensile strength (total plastic strain of 0.070 to 0.120 inch/inch) greatly reduced the time for failure by sulfide corrosion cracking. However, prestressing to below the ultimate tensile strength had little influence on the time to failure. Others[12] have reported that "the presence of plastic deformation accelerated the failure process for sulfide corrosion cracking." In the present work, the decreased cracking time of bolts prestressed beyond their ultimate tensile strength was not due to the decrease in cross sectional area resulting from prestressing. The decrease in area due to prestressing was a maximum of 10 percent, whereas the cracking time was reduced by a factor of 5-6.

Related Corrosion and Metallographic Studies

Corrosion of Type 4140 Steel in H_2S-H_2O Systems. Table 3 summarizes the corrosion rates for Type 4140 steel in the three test environments. The corrosion rates for long-time exposures (800-1800 hours) were usually less than those for short-time exposures (100-400 hours) due to the formation of a protective layer of sulfide scale. The rates for the 40 C—250 psi and the room temperature—atmospheric pressure systems were almost identical. A comparison of corrosion rates in the liquid phase of the three systems shows that the rates for the 120 C—250 psi environment were the highest for short exposure times and the lowest for long exposure times.

No apparent correlation existed between the corrosion rate in the three environments and the incidence of sulfide corrosion cracking. For example, the tendency for cracking was much lower in the 120 C environment, yet the corrosion rate for short-time exposures was the highest in this system. In addition, the corrosion rate for long-time exposures in the 120

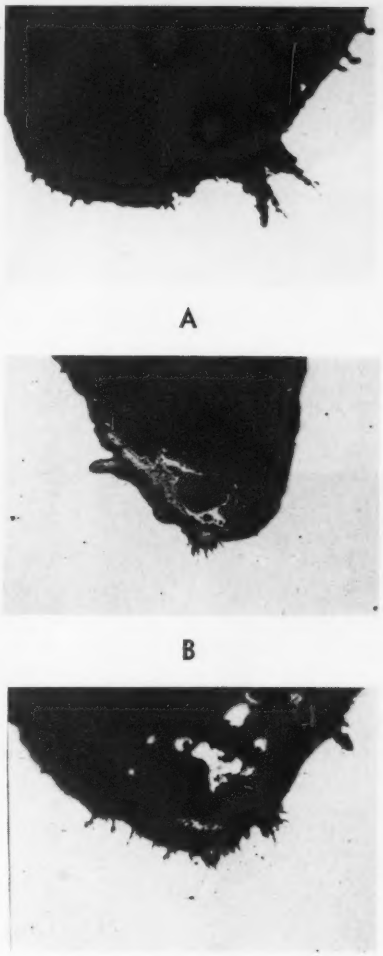


Figure 13—Microscopic pattern of sulfide corrosion at thread roots of Type 4140 bolts exposed in various H_2S-H_2O systems. All samples unetched. A: 40 C—250 psi system. Original magnification was 250X; magnification after reduction for engraving purposes was 225X. B: Room temperature—atmospheric pressure system. Original magnification was 100X; magnification after reduction for engraving purposes was 90X. C: 120 C—250 psi system. Original magnification was 150X; actual magnification after reduction for engraving purposes was 135X.

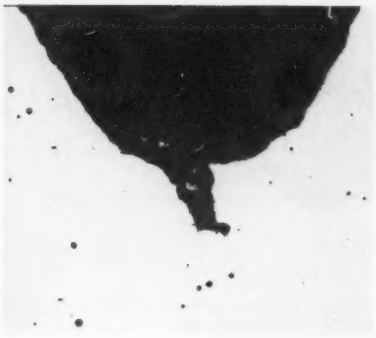
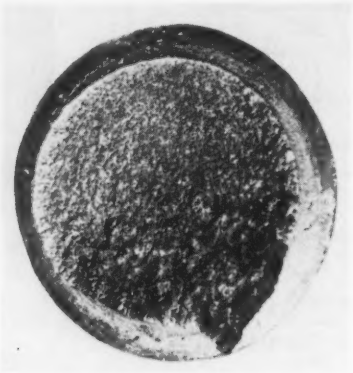
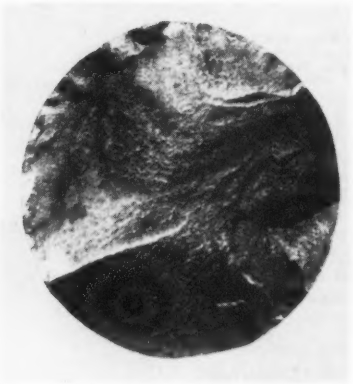


Figure 14—Large sulfide corrosion notch at thread root of Type 4140 bolt exposed in an H_2S-H_2O system at 40 C and 250 psi. Unetched, 150X.

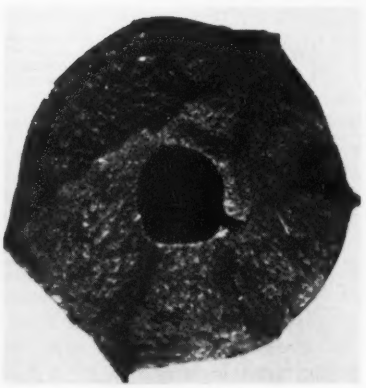


A—Gage failure. 6X

B—Nut failure. 6X



C—Shank failure. 6X



D—Head failure. 6X

Figure 16—Typical fractures of Type 4140 bolts which failed by sulfide corrosion cracking.

Figure 15—Large sulfide corrosion notches and accompanying crack at thread root of Type 4140 bolt exposed in an H_2S-H_2O system at room temperature and atmospheric pressure. Unetched, 100X (actual magnification after reduction for engraving purposes was 90X).

C system (liquid phase) was equal to those in the other two systems (vapor phase). It should be remembered that in the cracking tests the bolts were exposed in the liquid phase of the 120 C environment and the vapor phase of the other two environments.

The microscopic pattern of the corrosive attack was similar for all three test environments; this is shown in Figure 13. The localized attack caused by sulfide corrosion consisted of rounded pitting, blunt corrosion notches, and sharper notches approaching microcracks in appearance. It is easy to conceive how the very fine corrosion notches or microcracks shown in Figure 13 could develop into severe macrocracks under the influence of applied stress and the corrosive environment. Figures 14 and 15 illustrate the blunter type of corrosion notches; note the severe sulfide corrosion cracks originating from one of the notches.

The bolt hardness, applied stress level, and degree of plastic prestrain had very little effect on the type and extent of the corrosive attack observed on the bolts. As would be expected, the localized attack increased in both severity and frequency of occurrence as the exposure time increased. On the other hand, in exposures as short as 65 hours, a small amount of pitting generally occurred and occasionally, sharp corrosion notches or microcracks developed.

Fracture Studies on Type 4140 Bolt Failures. Sulfide corrosion cracking of the Type 4140 cap screws led to fractures at four principal locations: (1) in the threaded portion between the nut and the unthreaded shank (gage failure), (2) in the threaded portion engaged in the nut (nut failure), (3) in the unthreaded shank (shank failure), and 4) in the minimum cross section at the base of the cap (head failure). Representative macrographs of these four types of fracture are shown in Figure 16.

The typical gage failure began at a thread root and propagated through the cross section in a direction that was almost normal to the bolt axis. The result was an almost flat fracture except for a projection of torn metal marking the completion of fracture (see Figure 16A). Nut failures occurred in a similar fashion except that restraint by the nut threads caused multiple cracking. This is illustrated in Fig-

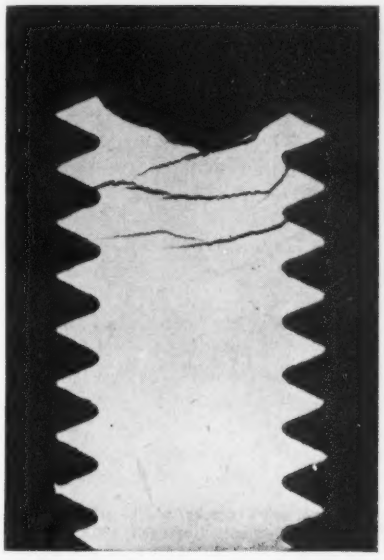


Figure 17—Sulfide corrosion cracks in longitudinal section of Type 4140 bolt which failed in the nut. Unetched, 3.5X.

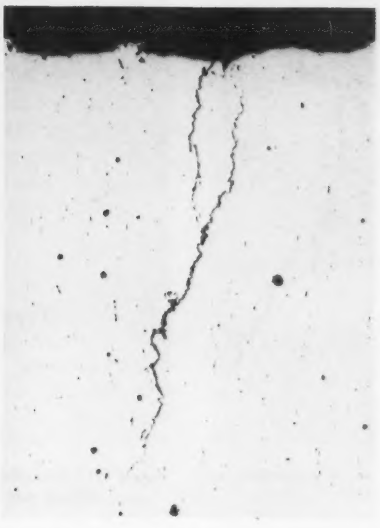


Figure 18—Sulfide corrosion cracks normal to fracture surface of failed Type 4140 bolt. Unetched, 100X.

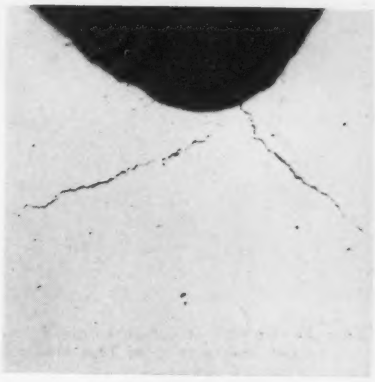


Figure 19—Fine sulfide corrosion cracks initiating at thread root of failed Type 4140 bolt. Unetched, 250X.

ure 16B where cracking has proceeded along three separate paths to fracture. The shank failures were always quite brittle in appearance due to irregular fracture contours (see Figure 16C). The head failures generally resulted from multiple cracking through the minimum cross section at the base of the socket in the bolt head (see Figure 16D).

There was no apparent correlation between the location of fracture and any of the variables involved in the sulfide corrosion cracking process. Even in ordinary bolting fractures where sulfide corrosion cracking is not involved, the fracture location will vary according to the stress concentration factors inherent in bolt design.[8] Thus, the lack of fracture correlation in the present study is not surprising.

Pattern of Macro- and Microcracking in Bolt Failures. Figure 17 illustrates the pattern of macrocracks in a bolt failure which occurred within the nut. Multiple macrocracks such as these were generally observed in most failures within the nut and occasionally in failures at other locations. Note that all of the cracks in Figure 17 initiated at points slightly removed from the thread roots. These points of crack initiation correspond roughly to the point of maximum stress concentration for loaded bolt threads engaged in a nut.[8] When bolt failures occurred in the gage, the cracks generally originated at the very root of the threads (see Figures 15 and 19).

Microscopic sulfide corrosion cracks similar to those shown in Figures 18-21 were often observed in failed bolts. These microcracks usually initiated at either the thread roots (Figure 19) or at the fracture surface (Figure 18). Quite often the cracks were of a branching nature (Figures 20 and 21) and, in many cases, they were filled with corrosion products (Figure 20).

TABLE 4—Effect of Metallic Coatings on the Susceptibility Of Type 4140 Bolts to Sulfide Corrosion Cracking

Environment	Type of Coating[1]	Number of Bolts Exposed[2]	Number of Bolts Failed	Average Time to Failure (Hours)	Exposure Time Unfailed Bolts (Hours)
H_2S-H_2O at 40C—250 psi	No coating	4	4	500	.....
	Cadmium	2	2	1,100	.....
	Zinc	2	2	1,100	.....
	Chromium	2	2	1,400	.....
	Lead	2	2	1,400	.....
	Brass	2	0	.....	8,200
	Nickel	4	0	.....	8,600
H_2S-H_2O at Room Temperature—Atmospheric Pressure	No coating	5	5	250	.....
	Zinc	2	2	500	.....
	Brass	2	2	600	.....
	Aluminum	3	2	1,700	11,000
	Cadmium	2	2	7,600	.....
	Lead	2	1	8,400	14,000
	Chromium	2	1	14,000	14,000

[1] All coatings were electrodeposits having a minimum thickness of 0.002 inches except the aluminum which was a sprayed paint coating applied in three successive light layers. The nickel plating was 0.005 inch thick.
[2] All bolts exposed had a hardness of Rc 38-42 and were stressed to 55-65,000 psi

Relationship Between Cracking and Microstructure. In most cases the sulfide corrosion cracks observed in failed bolts could not be classified as either intergranular or transgranular, because of the small and poorly defined grain size of the tempered martensite. However, some cracks did have a definite intergranular or transgranular appearance. Examples of these are shown in Fgures 22 and 23. There was no apparent relationship between the type of cracking observed and any of the test variables. Other investigators have reported sulfide corrosion cracking as being largely intergranular,[10] almost completely transgranular,[1,13] and sometimes intergranular and sometimes transgranular.[2,9,14] The results of the current study indicate that in a uniformly martensitic microstructure both types of cracking can occur.

Some of the previous studies of sulfide corrosion cracking emphasized the role of martensite in caus-

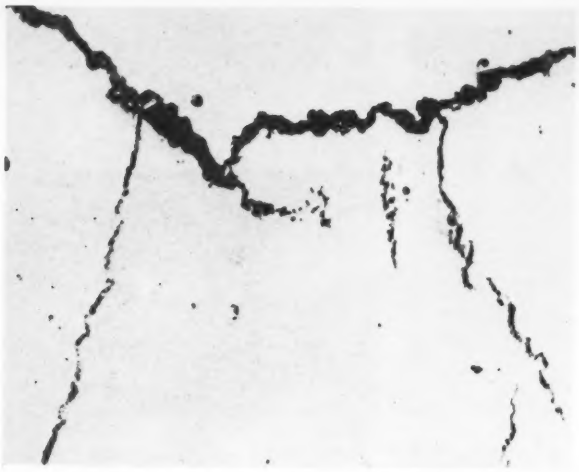


Figure 20—Network of sulfide corrosion cracks which originated at fracture surface of failed Type 4140 bolt. Unetched, 150X.

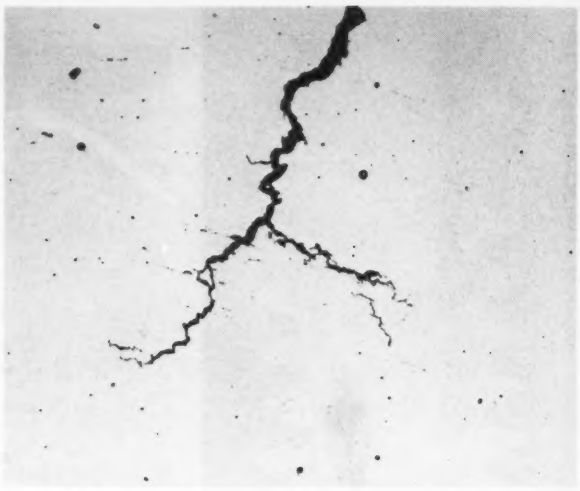


Figure 21—Branching sulfide corrosion cracks which originated at thread root of failed Type 4140 bolt. Unetched, 100X.

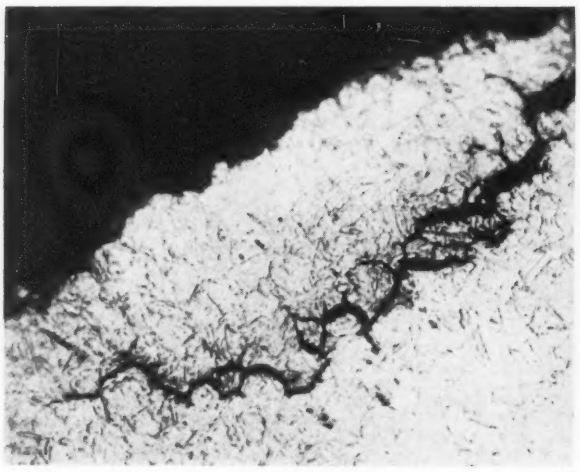


Figure 22—Sulfide corrosion crack with intergranular appearance in failed Type 4140 bolt. Etchant was 2 percent nital, 500X.

Figure 23—Sulfide corrosion crack with transgranular appearance in failed Type 4140 bolt. Etchant was 2 percent nital, 500X.

ing cracking.[12,13] These studies were primarily concerned with steels having a duplex microstructure containing varying amounts of untempered martensite. An examination of the bolts tested in this study revealed no microstructural change that could account for the sharp increase in cracking observed as the bolt hardness increased from R_c 25 to R_c 35. This is borne out by the series of photomicrographs in Figure 24.[6] The only microstructural change evident in these pictures is the increased spheroidization of carbides that occurs as the hardness is reduced from R_c 55 to R_c 21 by tempering. In addition, there was no microscopic evidence of untempered martensite resulting from decomposition of retained austenite in any of the bolts.[7]

[6] Figures 24A and 24B have been included to show the marked difference between a ferritic-pearlitic microstructure and a tempered martensitic microstructure of the same hardness.

[7] Quenching of hardened and tempered bolts in liquid nitrogen did not produce any increase in hardness. This indicated that retained austenite was not present in appreciable amounts.

Prevention of Bolting Failures in H_2S-H_2O Environments

Three methods of eliminating bolting failures due to sulfide corrosion cracking were studied: (1) use of metallic coatings on Type 4140 bolts, (2) addition of corrosion inhibitors to the H_2S-H_2O systems, and (3) use of other bolting materials in place of Type 4140 steel.

Effect of Metallic Coatings. Seven different types of metallic coatings were applied to Type 4140 bolts prior to their exposure in two different H_2S-H_2O systems. The hardness and stress level of these bolts were sufficient to ensure the cracking of uncoated bolts. Table 4 gives the results of this study.

While most of the coatings were effective in increasing bolt life by a factor of 2-3 or more, they did not provide consistent or reliable protection against sulfide corrosion cracking in both test environments.

The relatively heavy nickel plating (0.005 inch thick) gave good protection in the one environment in which it was tested. Others[15] have reported that electroplated coatings of nickel can be used to prevent sulfide corrosion cracking.

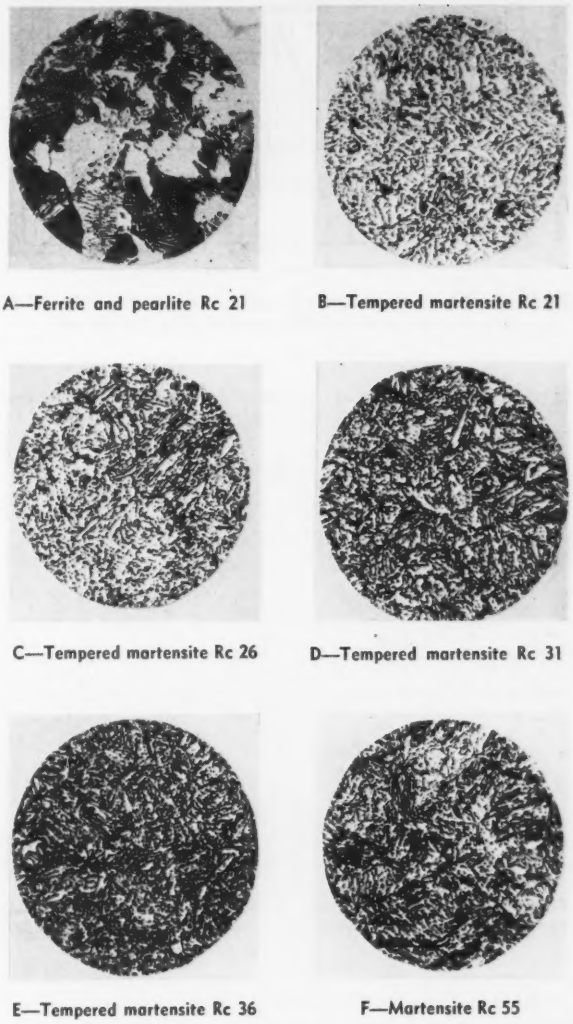
Despite its effectiveness in these tests, nickel plating was rejected as a method of preventing bolting failures in the field. This was done because the making and breaking of threaded connections materially increases the probability of mechanical rupture of the coating.

Effect of Inhibitors. Two inhibitors were evaluated in this study: Duomeen T[8] and tetraethylenepentamine (TEPA). These two low-vapor-pressure amines had proven effective in reducing the general corrosion of steel in H_2S-H_2O systems. For example, the addition of either of these inhibitors to the liquid phase of the 40 C—250 psi system reduced the corrosion rate for short-time exposures (100-400 hours) from 9 to 1.5 mil/yr. The cracking tests were made in the liquid phase of pressurized H_2S-H_2O systems at 40 C with about 500 ppm. of the inhibitor added to the liquid phase. The hardness and stress level of the test bolts were sufficient to cause cracking in the absence of any inhibitor.

Figure 25 compares the cracking behavior of Type 4140 bolts in the two inhibited systems with that in the uninhibited system. The tetraethylenepentamine (TEPA) proved the more effective of the two inhibitors in these tests because it reduced the percentage of bolt failures from 100 percent to 40 percent. The practical significance of these tests is limited for two reasons: (1) the number of bolts tested was insufficient for a reliable evaluation and (2) neither inhibitor was 100 percent effective in preventing failure. However, the data do reflect the findings of others[1,15] who used inhibitors to combat sulfide corrosion cracking with varying degrees of success.

Evaluation of Substitute Materials. As shown by the data in Tables 5 and 6, 5 percent aluminum bronze and 75ST aluminum were the only substitute bolting materials tested which showed any susceptibility to sulfide corrosion cracking. Even there, only one bolt of each material failed and then only after a long exposure (1 to 1½ years). Type 316 stainless steel (annealed condition), K-Monel, Inconel, and Inconel X proved to be completely resistant to cracking in the two H_2S-H_2O systems investigated. K-Monel and Inconel X were immune to sulfide corrosion cracking even at high levels of hardness and applied stress, R_c 32-36 and 100-125,000 psi, respectively. The above results confirm the excellent resistance to sulfide corrosion cracking reported in the literature for annealed Type 316 stainless steel,[9] Monel, K-Monel, and Inconel.[1,3,12]

Visual observations indicated that Inconel, Inconel X, and Type 316 stainless steel had excellent resistance to general corrosion in those H_2S-H_2O systems



A—Ferrite and pearlite Rc 21 B—Tempered martensite Rc 21

C—Tempered martensite Rc 26 D—Tempered martensite Rc 31

E—Tempered martensite Rc 36 F—Martensite Rc 55

Figure 24—Microstructure of Type 4140 bolts as a function of hardness. Etchant was 2 percent nital, 1000X.

tested. Monel and K-Monel were slightly corroded in the same systems, while 5 percent aluminum bronze was severely corroded. The corrosion resistance of 75ST aluminum was quite poor, mainly because of iron sulfide scale in the test system.

The only microcracks observed in the failed 75ST aluminum and 5 percent aluminum bronze bolts were located adjacent to the fracture surface. The cracking in the 75ST aluminum bolt was clearly intergranular (see Figure 26). In the 5 percent aluminum bronze bolt, the cracking appeared to be at least partially intergranular (see Figure 27). Both of these bolt failures, in contrast to the Type 4140 bolt failures, were accompanied by severe general corrosion.

Mechanism of Sulfide Corrosion Cracking

Throughout this paper the term "sulfide corrosion cracking" has been used to describe the type of failures which occur in H_2S-H_2O environments. This term arose from the lack of clearcut evidence as to whether sulfide corrosion failures were the result of stress corrosion cracking or hydrogen embrittlement.[1]

[8] Duomeen T is an aliphatic amine type of commercial inhibitor marketed by Armour and Co.

[9] It is recognized that Type 316 stainless steel is susceptible to sulfide corrosion cracking in the cold-worked condition.

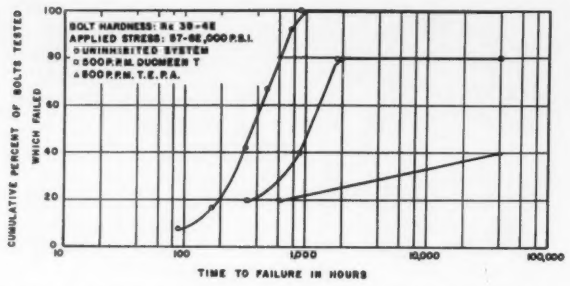


Figure 25—Effect of inhibitors on cracking susceptibility of Type 4140 bolts exposed in an H_2S-H_2O system at 40 C and 250 psi.

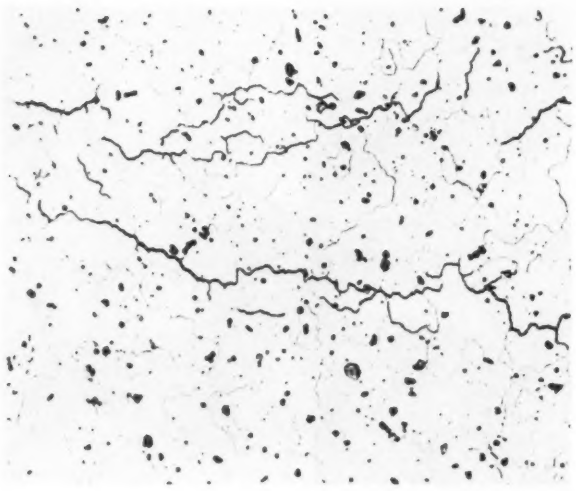


Figure 26—Intergranular sulfide corrosion cracks near the fracture surface of failed 75ST aluminum bolt. Etchant was 0.5 percent HF, 250X.

Such uncertainty stems from the very manner in which ferrous materials corrode in aqueous hydrogen sulfide.

Sulfide Corrosion Reaction. Hydrogen sulfide when dissolved in water forms a weak acid which can attack steel surfaces and thereby release hydrogen according to the following equation:

$$Fe + H_2S \rightarrow FeS + 2H \rightarrow FeS + H_2$$

By "poisoning" the surface available for the recombination of atomic hydrogen into molecular hydrogen, the H_2S acts as a promoter for the entry of atomic hydrogen into the steel.[3] The blisters found in soft steels exposed to sulfide environments offer striking proof that the sulfide corrosion reaction is capable of charging a steel with hydrogen.[3] Analysis of the gas contained in such blisters showed it to be 99.5 percent hydrogen.[3]

Prior Theories of Cracking. Previous investigators, in their proposed mechanisms of sulfide corrosion cracking, have been divided between stress corrosion cracking and hydrogen embrittlement. Some have attributed sulfide corrosion failures solely to stress corrosion cracking.[2,5,6] Certainly, the phenomenon of sulfide corrosion cracking has present all of the elements required for stress corrosion cracking. These

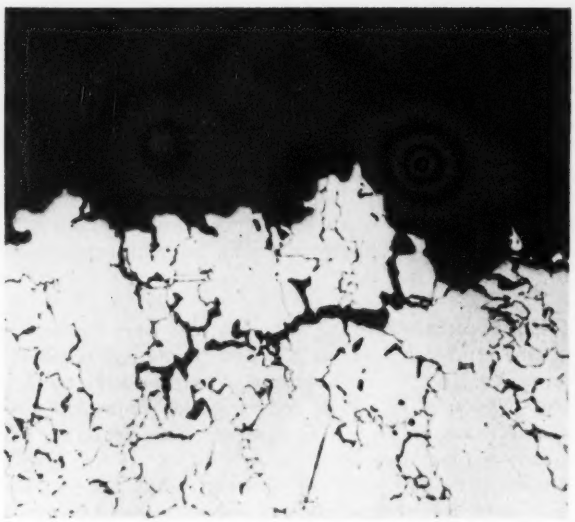


Figure 27—Sulfide corrosion cracks adjacent to fracture surface of failed 5 percent aluminum bronze bolt. Etchant was NH_4OH-H_2O_2. 250X.

elements consist of an applied tensile stress and a corrosive environment acting together to produce failure in a more or less spontaneous fashion.

Other investigators have felt that the failures in H_2S-H_2O environments were due directly to hydrogen embrittlement.[3,4,16,17] Hafsten,[4] for example, found a good correlation between the degree of hydrogen embrittlement and the cracking of carbon steel springs in an 8-10 percent H_2S atmosphere. One of these springs after having failed in service was quickly sectioned into three parts. One part was immediately subjected to a simple bend test and failed in a completely brittle fashion. The other two sections, one of which was baked for 4 hours at 400 F and one of which was aged for three weeks at room temperature, failed in a ductile fashion after undergoing appreciable plastic deformation. Prange[17] has pointed out that those sulfide environments which cause brittle failures in hardened steels all produce blistering in soft steel. The latter, of course, can be directly attributed to hydrogen.

Fraser and Treseder,[1] while favoring stress corrosion cracking as the primary mechanism of sulfide corrosion failures, have recognized the role played by hydrogen embrittlement. They have proposed a combined mechanism in which (1) a very small crack is initiated through stress corrosion and (2) the crack is propagated by additional stress caused by the precipitation of hydrogen just ahead of the crack.

Interpretation of Current Data. It is believed that the sulfide corrosion cracking data presented in this paper are best explained on the basis of hydrogen embrittlement. From this viewpoint, the primary role of the sulfide corrosion reaction was to charge the steel with hydrogen and to produce pits or notches which could serve as initiation sites for cracks. The contribution of hydrogen embrittlement to failure by sulfide corrosion cracking is emphasized by three factors: (1) the decrease in cracking tendency with in-

creased test temperature, (2) the correlation between hardness and susceptibility to cracking, and (3) the similar cracking behavior of bolts exposed to H_2S-H_2O systems and inhibited acid pickling solutions.

Effect of Temperature on Cracking Tendency. As pointed out earlier, the greatly reduced cracking tendency in the 120 C test system could not be explained on the basis of either a reduced corrosion rate or a change in the pattern of corrosive attack. However, the reduced cracking tendency at 120 C can be explained on the basis of hydrogen embrittlement as the primary cause of cracking. For example, it is known that the degree of embrittlement caused by hydrogen decreases with increasing temperature in the range of 0-100 C.[18, 19, 20]

The effect of test temperature on the hydrogen embrittlement of hardened steels is best shown by the work of Hobson and Hewitt.[20] They ran tensile tests on specimens of a 3 percent Cr-Mo steel (0.28 percent C) which had been charged with hydrogen by a high-pressure heating technique. Reduction in area was used as a measure of the degree of embrittlement. Figure 28 shows the results of tests made at 20 and 100 C for three levels of hardness.[10] Note that at a hardness of R_c 46 the steel was significantly embrittled at both 20 C and 100 C. This could explain why cracking of R_c 40 bolts was observed in sulfide environments at both room temperature and 120 C. Figure 28 also shows that at a hardness of R_c 32 the degree of embrittlement at 100 C was appreciably less than at 20 C. This might explain why R_c 30 bolts frequently cracked in sulfide environments at room temperature but never at 120 C.

Correlation of Hardness with Susceptibility to Cracking. The susceptibility of Type 4140 bolts to sulfide corrosion cracking was found to be directly related to the hardness or strength level of the bolts (see Figures 5, 6, 7, 8, and 10). The strength level (or hardness) to which a steel is heat treated also has a similar strong effect on its susceptibility to hydrogen embrittlement.[17, 21, 22, 23]

[10] Data were taken from Figure 3 of Reference No. 20. Data for a hardness of R_c 11 are not included for two reasons: (1) prolonged tempering was used to obtain such a low hardness and (2) the results obtained at 20 C for a 3 percent Cr-Mo steel of R_c 11 hardness did not agree with previously reported results for other steels at the same strength level (see Figure 13 of Reference No. 21). The latter observation indicated that at these low strength levels some embrittling effect other than hydrogen was influencing the ductility of the 3 percent Cr-Mo steel.

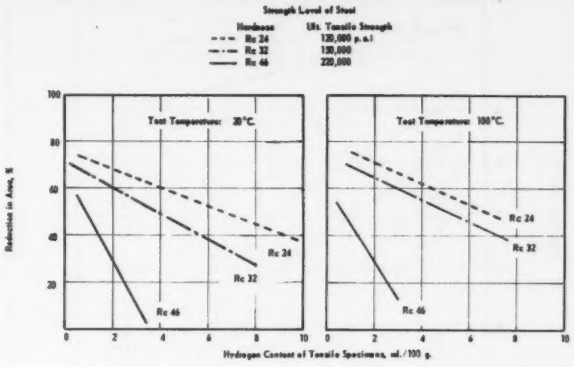


Figure 28—Effect of testing temperature on hydrogen embrittlement of a 3 percent Cr-Mo steel.[20]

Hobson and Sykes[21] have shown by means of tensile tests that the reduction in area of hydrogen-charged steels decreased rapidly as the strength level was increased. Their data[11] for a 3 percent Cr-Mo

[11] Data were taken from Figure 9 of Reference No. 21. Data for the 41-47 tsi strength levels were omitted for the reason given in a previous footnote.

TABLE 5—Summary of Test Data for Substitute Bolting Materials Exposed in an H_2S-H_2O System at 40 C and 250 psi

Bolting Material[1]	Average Physical Properties of Bolts[2]			Range of Applied Stress, psi	Number of Bolts Tested	Number of Bolts Failed	Total Exposure Time, Months
	Rockwell Hardness	Yield Strength, psi (0.2% Offset)	Ultimate Tensile Strength, psi				
Type 316 Stainless Steel	R_b 86	35,000	90,000	(45-85,000)[3]	19	None	12
K-Monel	R_c 22	105,000	125,000	65-90,000	5	None	11
	R_c 33	135,000	160,000	100-125,000	2	None	4
Inconel	R_c 28	130,000	140,000	90-110,000	8	None	11
Inconel X	R_c 20	100,000	110,000	100,000	1	None	12
	R_c 35	135,000	180,000	100-110,000	4	None	9
5% Aluminum Bronze	R_b 90	65,000	95,000	(50-95,000)[3]	6	None	11

[1] Test specimens were ⅜—½ inch in diameter stud bolts.

[2] Strength values for Type 316 stainless steel bolts of R_b 86 hardness were obtained from the literature.

[3] These values of applied stress were calculated on the basis of Hooke's law even though the yield point of the bolts had been exceeded. Consequently, these stresses are not representative of the actual bolt stresses, but are indicative of the total strain introduced in stressing.

TABLE 6—Summary of Test Data for Substitute Bolting Materials Exposed in an H_2S-H_2O System at Room Temperature and Atmospheric Pressure

Bolting Material[1]	Average Physical Properties of Bolts[2]			Range of Applied Stress, psi	Number of Bolts Tested	Number of Bolts Failed	Total Exposure Time, Months
	Rockwell Hardness	Yield Strength, psi (0.2% Offset)	Ultimate Tensile Strength, psi				
K-Monel	R_c 22	105,000	125,000	70-90,000	5	None	22
	R_c 33	135,000	160,000	105-125,000	2	None	12
Inconel	R_c 28	130,000	140,000	80-110,000	8	None	22
Inconel X	R_c 20	100,000	110,000	90-95,000	2	None	18
	R_c 35	135,000	180,000	100-125,000	4	None	18
5 Percent Aluminum Bronze	R_b 90	65,000	95,000	50-60,000	5	1	21
				(65-75,000)[3]	2	None	21
75ST Aluminum	R_b 90	80,000	90,000	60-70,000	2	1	19
				(110,000)[4]	1	None	19

[1] Test specimens were ⅜—½ inch in diameter stud bolts.

[2] Strength values for 75ST aluminum bolts of R_b 90 hardness were obtained from the literature.

[3] These values of applied stress were calculated on the basis of Hooke's law even though the yield point of the bolts had been exceeded. Consequently, these stresses are not representative of the actual bolt stresses, but are indicative of the total strain introduced in stressing.

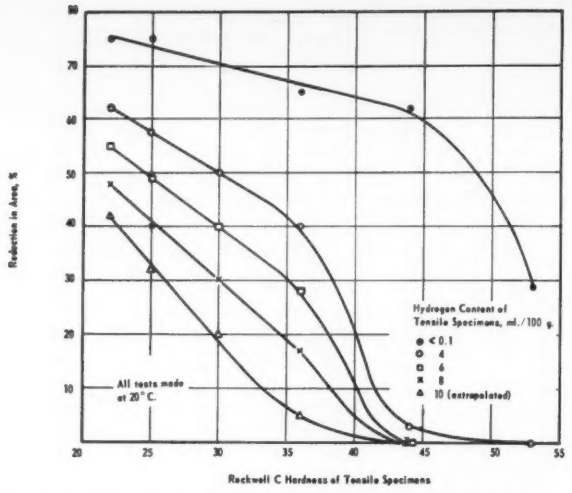


Figure 29—Effect of hardness on hydrogen embrittlement of a 3 percent Cr-Mo steel.[23]

TABLE 7—Cracking of Type 4140 Bolts in an Inhibited Hydrochloric Acid Pickling Solution*

Hardness Range	Range of Applied Stress, psi	Number of Bolts Pickled	Number of Bolts Failed
R_c 40-42	60-70,000	10	10
R_c 39-43	20-35,000	10	0
R_c 30-34	105-120,000	10	4
R_c 30-34	50-70,000	16	0

* All bolts were picked in place on test bars for 2 hr. in a pickling solution consisting of 10% HCl (by weight) and 0.2% of Barrett Pickling Compound No. 53 (by volume). About 100 g. of sulfide scale was added to the system to act as a poison.

steel (0.26 percent C) have been replotted in Figure 29 for five levels of hydrogen content. (The specimens were charged with hydrogen by both electrolytic and high-pressure heating techniques.) Note the sharp decrease in reduction in area that occurred as the hardness was increased from R_c 35 to R_c 45.

The curve in Figure 29 for a hydrogen concentration of 10 ml/100 g is particularly pertinent in the case of the bolting study. The hydrogen content of steel specimens exposed to a pressurized H_2S-H_2O system at 40 C for two weeks averaged between 9 and 11 ml/100 g. (These values represent the amount of hydrogen collected at room temperature over mercury immediately following the test exposure.) Note the similarity in the shape of the 10 ml/100 g curve in Figure 29 and the cracking curve in Figure 6. The rapid decrease in reduction in area in Figure 29 between R_c 30 and R_c 40 appears to parallel quite closely the increase in cracking susceptibility in Figure 6 between R_c 27 and R_c 40.

Parallel Cracking Behavior of Bolts in Sulfide Environments and in Pickling Solutions. Pickling in acid solutions is known to cause hydrogen embrittlement of both mild and hardened steels.[22,24] To determine the effect of hydrogen embrittlement on hardened and drawn Type 4140 bolts, a series of bolts were exposed under stress to an inhibited 10 percent hydrochloric acid solution for two hours. Sulfide

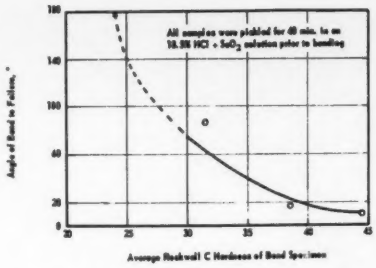


Figure 30—Effect of hardness on hydrogen embrittlement of a 12 percent Cr steel.[22]

scale was added to the pickling solution to promote the entry of hydrogen into the bolts. The data in Table 7 show that the cracking behavior of the bolts in the inhibited acid pickling solution was similar in two respects to the cracking behavior in H_2S-H_2O systems. First, the harder bolts were more susceptible to cracking; 100 percent of the R_c 40 bolts failed at 60-70,000 psi while none of the R_c 30 bolts failed at that stress level. Secondly, increasing the applied stress increased the susceptibility to cracking. For example, at each of the two hardness levels there was a definite stress level below which no cracking occurred.

Durkin[22] also has shown that the degree of hydrogen embrittlement caused by acid pickling solutions is directly related to the hardness level. He exposed 12 percent Cr steel samples of varying hardness to an 18 percent HCl pickling solution to which SeO_2 was added as a poison. Constant rate bend tests were then made on the specimens to determine the loss in ductility due to hydrogen. Durkin's data for a 40 minute exposure in the acid pickling solution have been replotted in Figure 30. The angle of bend to failure clearly decreased with increasing hardness. Note that at a hardness of R_c 25 the specimen bent through an angle of 180 degrees without failure. The curve in Figure 30 for hydrogen embrittlement due to acid pickling is quite similar in appearance to that in Figure 6 for sulfide corrosion cracking.

Summary

1. Failure of Type 4140 bolts by sulfide corrosion cracking was observed in H_2S-H_2O systems at temperatures of 20 C to 120 C and at pressures of 1 to 17 atmospheres. The susceptibility to cracking increased with increasing bolt hardness, increasing applied stress, increasing amounts of plastic deformation, and decreasing test temperature.

   a. In the absence of plastic deformation, no bolt failures occurred at hardnesses below R_c 27 even under stresses approaching the yield strength of the material. In the hardness range of R_c 27-55, the cracking susceptibility increased with increasing hardness and increasing applied stress. For each hardness

level, there appeared to be a minimum or threshold stress required to produce cracking. Increasing the hardness lowered the minimum stress for cracking.

b. Plastic prestraining of the bolts (1) lowered the minimum hardness for sulfide corrosion cracking from R_c 27 to R_c 22 and (2) increased the number of bolt failures in the hardness range of R_c 27-33.

c. Increasing the test temperature from 40 C to 120 C greatly reduced the tendency for sulfide corrosion cracking. The pressure of the test system, in the range of 1-17 atmospheres, had little effect on the occurrence of cracking.

d. The time for failure by sulfide corrosion cracking decreased sharply with increasing hardness and decreased to a much lesser degree with increasing applied stress and increasing amounts of plastic deformation.

2. The use of protective metallic coatings and the addition of corrosion inhibitors to the test system were evaluated as means of preventing the sulfide corrosion cracking of Type 4140 bolts.

a. An electroplated coating of nickel (0.005 inch thick) provided adequate protection against sulfide corrosion cracking. Other types of electroplated coatings such as Cd, Zn, Pb, Cr and brass did not give reliable or consistent protection.

b. Addition of inhibitors to the liquid phase of the test system reduced, but did not eleminate, the failure of bolts exposed in the liquid phase.

3. The following bolting materials were completely resistant to sulfide corrosion cracking under those conditions tested: Inconel, Inconel-X, K-Monel, and annealed Type 316 stainless steel. Type 75ST aluminum and 5 percent aluminum bronze were susceptible to a minor degree to stress corrosion cracking in one H_2S-H_2O system.

4. The failure of Type 4140 bolts in aqueous H_2S systems appears to be best explained on the basis of hydrogen embrittlement. The sulfide corrosion reaction serves to charge the steel with hydrogen and to create pits or notches which act as sites for crack initiation.

Acknowledgments

Particular thanks are due W. J. Evans who assisted with most of the experimental work, W. B. DeLong who was in charge of the overall experimental program on sulfide corrosion, and J. A. Snyder who was responsible for the general corrosion studies.

The information included in this paper was developed during the course of work under contract A.T. (07-2)-1 for the Atomic Energy Commission, whose permission to publish is gratefully acknowledged.

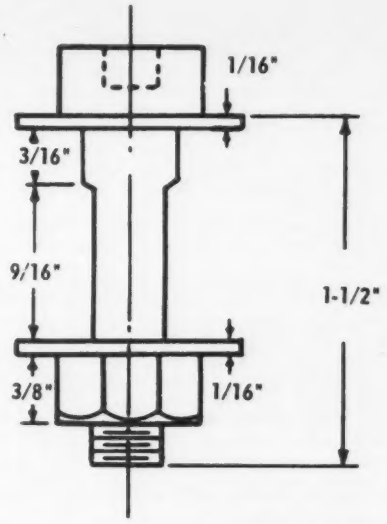


Figure 31—Sketch of 3/8-inch Allen head cap screw.

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APPENDIX

The derivation of the formula used for calculating stress on ⅜ inch Allen head cap screws from measused values of elongation is given below. A sketch of the Allen head cap screw may be found in Figure 31.

$$\text{Hooke's Law: } E = \frac{\text{Stress}}{\text{Strain}} = \frac{P/A}{e/L} \quad (1)$$

where P = load, A = area, e = elongation, L = gage length over which elongation occurs, and E = modulus of elasticity.

Solving Equation (1) for e: $e = \frac{PL}{EA}$ (2)

Let: e_u = elongation of unthreaded shank of bolt
e_t = elongation of threaded region within gage length of bolt (gage length included one-half of the nut thickness).
e = total elongation of bolt

Then: $e = e_u + e_t$ (3)

Substituting Equation (2) in Equation (3):

$$e = \frac{PL_u}{EA_u} + \frac{PL_t}{EA_t} = \frac{P}{E}\left(\frac{L_u}{A_u} + \frac{L_t}{A_t}\right) \quad (4)$$

where L_u = length of unthreaded shank = 0.250 in.
L_t = length of threaded region = 0.812 in.
A_u = area of unthreaded shank = 0.111 sq. in.
A_t = minimum area of threaded region = 0.0701 sq. in.

Substituting Numerical Values in Equation (4):

$$e = \frac{P}{30 \times 10^6}\left(\frac{0.250}{0.111} + \frac{0.812}{0.0701}\right) = 4.62 \times 10^{-7}P \quad (5)$$

$$P = 2.16 \times 10^6 e \quad (6)$$

$$\text{Maximum stress in bolt: S max.} = \frac{P}{A\ (\text{min.})} = \frac{P}{0.0701} \quad (7)$$

Substituting Equation (6) in Equation (7):

$$S\ \text{max.} = \frac{2.16 \times 10^6 e}{0.0701} = 3.08 \times 10^7 e \quad (8)$$

Maximum stress in bolt = (3.08×10^7) (measured elongation)

Any discussions of this article not published above will appear in the December, 1957 issue.

Thermoplastic Materials for Pipe*

By P. M. ELLIOTT

Introduction

THE PRODUCTION and installation of pipe is big business. At the present time more than nine million tons of ferrous pipe and tube alone are sold annually. It has been estimated that the annual bill for corrosion of pipe is a staggering six hundred million dollars. It is small wonder, then, that in recent years much attention has been paid to new materials for the production of pipe.

Plastics with their low specific gravity, generally good chemical resistance, good electrical properties, and general ease of handling are obvious raw materials for pipe. It is not surprising that during the last decade ever increasing amounts of plastic materials have been going into pipe production.

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Abstract

The general properties of plastic pipe are outlined and comparisons made with metal pipe. Particular attention is given to polyethylene, acrylonitrile copolymer blends, cellulose acetate butyrate, polvinyl chloride, and polyvinylidene chloride. Heat distortion temperatures and range of recommended temperatures are given for these materials. Data showing rate of testing versus fiber stress at burst, fiber stress versus time to fail, hoop stress versus service life, tensile stress versus time to rupture, fiber tress at burst versus temperature, tensile strength versus temperature and safe hoop stress versus temperature are given for some of these plastics. The joining of plastic pipe is discussed briefly. Other topics considered include long term effects of stresses, temperature effects, and new developments in plastic piping. 7.2

Thermoplastics Used in Pipe

The currently important thermoplastics for pipe production are polyethylene, acrylonitrile copolymer blend compounds, cellulose acetate butyrate, polyvinyl chloride, and polyvinylidene chloride. It is very difficult to obtain accurate estimates of the amounts of these materials that have been used in pipe, but published figures seem to divide the market much as indicated in Table 1. The relative importance of the two types of materials has changed during this period, but during the last five years polyethylene has constituted a major portion of the total thermoplastic used. It is expected that large amounts of polyethylene will continue to be used, but the special properties of other materials—notably polyvinyl chloride and acrylonitrile copolymer blend materials are expected to increase their share of the total market.

General Properties of Plastic Pipe

Advantages

As a class, plastic materials have certain general advantages relative to the metals commonly used for pipe production. Plastics have considerably lower density than even aluminum; hence, plastic pipe is light in weight, which makes it easy to handle, easy to ship, and easy to store. The generally good electrical properties of plastic materials eliminate the electrolytic effects which are proving so troublesome in many pipe installations. Even metals that are not attacked by direct corrosive action may be damaged by electrolysis. Plastics in general are not subject to "corrosion." Resistance to acids, alkalies, salts, and oxidizing media vary widely from material to material, but many of the plastic pipe materials have good general chemical resistance. This is of importance from the consideration not only of the materials being carried within the pipe but also of the external surroundings. Many soils and atmospheres have proven highly corrosive to metallic pipe.

Plastics, in general, have little or no tendency to introduce contamination into materials being transported. In metal pipes, rates of solution which may not be serious from the point of pipe corrosion may still be detrimental because of the catalytic effects of many metal ions even at low concentrations.

From the standpoint of long-term economy, plastics frequently have an advantage over metal pipe. Even in cases where the initial per foot cost of the plastic pipe may be higher than for some other types of pipe, ease of installation and long life expectancy of plastic pipe add up to a more economical installation than a corresponding metal piping system.

Limitations

It must be kept in mind that plastic piping has certain definite limitations. The thermoplastic pipe materials in general have considerably lower tensile

★ Submitted for publication December 19, 1956. A paper presented at a meeting of the Western Region, National Association of Corrosion Engineers, Long Beach, Califorina, November 16, 1956.

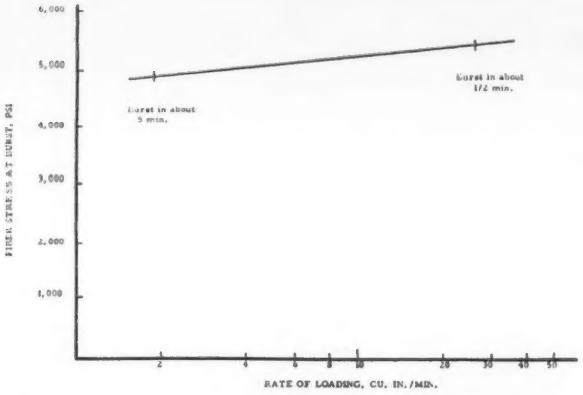

Figure 1—Rate of testing versus fiber stress at burst for acrylonitrile copolymer blend pipe.

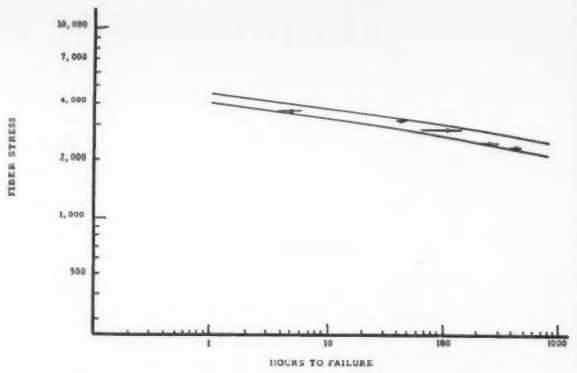

Figure 2—Fiber stress versus time to fail for 1½-inch acrylonitrile copolymer blend pipe at 73 F.

TABLE 1—Estimated Production of Thermoplastic Pipe in Millions of Pounds

ITEM	YEAR						
	1950	1951	1952	1953	1954	1955	Est. 1956
Flexible Pipe	2.0	4.5	9.0	23.0*	32.0*	42.0	(50)
Rigid Pipe	3.3	5.0	5.5	6.5	8.5	13–14	(18–19)
Total	5.3	9.5	14.5	29.5	40.5	45–46	(68–69)

*Includes scrap estimated at 5 million in 1953; 10 million in 1954; 12 million in 1955.

strength than the metals. As a result, plastic piping is limited to low or moderate pressure installations.

The fact that thermoplastics soften when heated means that they lose strength as temperature is raised; hence the upper temperature limit of serviceability is relatively low. Even the best present thermoplastic pipe usually is not recommended for use at temperatures above 170 F in applications where there is much, if any, internal pressure. A few experimental installations have been made at temperatures as high perhaps as 220 F but this is certainly an upper limit for current pipe materials.

As mentioned above, the chemical resistance of each material is an inherent characteristic. The choice of pipe material will be dictated by the chemicals with which it is to come in contact and must be the subject of careful study. In many cases, the material manufacturer and the pipe producer will not have definite information on the resistance of a given pipe material to a specific set of corrosive conditions. It then becomes necessary for the purchaser of the pipe to make a test installation or to take a calculated risk on the basis of the information which is available.

The moderate stiffness of thermoplastic pipe materials plus the fact that they are subject to flow when stressed, particularly if the temperature is high, means that extra care must be taken in the support of above ground installations. Another factor which has frequently been overlooked and which has caused failure in some pipe installations is the relatively high coefficient of thermal expansion of plastic materials. Most thermoplastics have coefficients of thermal expansion at least five times that of steel and, for some materials, the difference may be as high as fifteen times.

Joining of Plastic Pipe

The lack of availability of suitable fittings and valves for use with plastic pipe has been and continues to be a problem in the growth of many applications. In the early application of plastics in the piping field in this country, much flexible material was used. The pipe was made available in long coiled lengths which, among other things, decreased the number of fittings required in any given installation. Metal insert fittings and metal clamps were used to fasten together the individual lengths of pipe. As suitable injection molding compounds became available, plastic fittings were developed and most flexible pipe is now joined with plastic insert fittings. Metal clamping bands are still commonly used.

For the joining of rigid pipe, several methods have been employed. For the heavier wall IPS schedule 120 and IPS schedule 80 and to some extent IPS schedule 40 pipe made with acrylonitrile copolymer blends, Type II PVC and polyvinylidene chloride standard threaded fittings have been used. It has been found that ordinary pipe threading equipment used with reasonable care will form suitable threads on these pipes. In recent months there has been a trend away from the use of threaded fittings with the thinner wall IPS schedule 40 type pipe. The threaded fittings have been used most extensively in chemical plant installations of pipe where frequently there are relatively complex installations requiring many fittings.

A second method of joining rigid pipe is by solvent or cement welding. A completely different schedule of pipe sizes designated as SWP (solvent weld pipe) has been developed for this system of joining. This new schedule of sizes was designed primarily to prevent ill-advised attempts at threading since, in general, the wall thickness is less than the IPS schedules suitable for threading. SWP pipe has been widely used with cellulose acetate butyrate, acrylonitrile copolymer blends, and both types of PVC. At the present time, non-threaded fittings suitable for use with IPS pipe are being produced and the solvent

weld technique is being used with the thinner wall IPS schedule 40 and schedule 20 pipe.

Welding has been used as a method of joining Type I PVC and polyvinylidene chloride pipe. The technique has been employed extensively in Europe, particularly for the joining of large diameter PVC pipe or for the fabrication of fittings and special shapes. There has been less wide-spread use of the technique in this country, except for the production of special fittings and valves.

At the present time, several manufacturers are furnishing fittings and valves made from all the common pipe materials with the exception of polyethylene. Most of these fittings are injection molded. To indicate the complexity of the problems of the fitting manufacturer, one has merely to note that one manufacturer lists over 600 types and sizes of SWP and IPS fittings for each of three different materials.

For use with SWP pipe, extruded couplings have been employed. Since these couplings in general are not tapered, considerable trouble has been experienced with the fit of such joints. As a result most manufacturers are looking with increased favor toward injection molded fittings. Valves, either fabricated or molded in accordance with special patented designs, are available in a wide range of sizes for use with most of the rigid pipes.

Long-Term Effects

It has long been known, although apparently sometimes overlooked, that materials cannot sustain for long times the stress levels determined in short term tensile tests. At stresses considerably below these "tensile" figures, the materials are subject to creep or may even fail by complete rupture. Of importance for any application involving continued stress is a knowledge of how high a stress can be applied to a given material without causing undue creep and/or rupture. A self-explanatory name which has been applied to this maximum stress level is "the long-term effective tensile."

To illustrate the derivation and significance of long-term effective tensile figures, consider a series of tests run on acrylonitrile copolymer blend materials. Figure 1 illustrates the effect of rate of testing on the tensile strength of one of these compounds. If pressure in the pipe is increased rapidly enough to bring about burst in about ½ minute, the fiber stress at burst is 5400 psi. If pressure increase is less rapid, so that the duration of test is about five minutes, the fiber stress at burst is about 4900 psi. It is obvious that extension of such a test to a point to where burst would take place in, for example, ten years would give a figure considerably below this level.

No method is available to the author to determine what stress could be supported for ten years, other than to stress pipe at varying levels and hold it under pressure to see how long it will last. Figure 2 shows data obtained in such a series of tests on pipe made with acrylonitrile copolymer blends. The short horizontal lines indicate the spread of data obtained at replicate tests. The long parallel lines on the

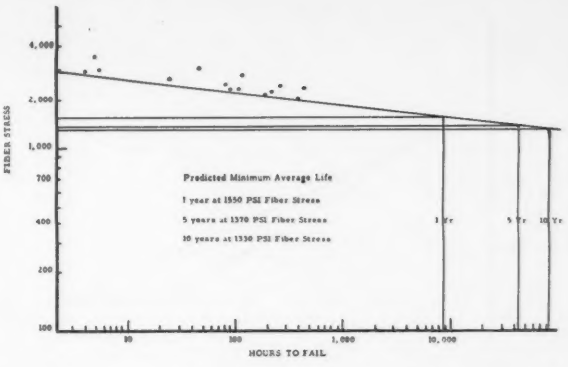

Figure 3—Extrapolation of data showing fiber stress versus time to fail for 1½-inch acrylonitrile copolymer blend pipe at 73 F.

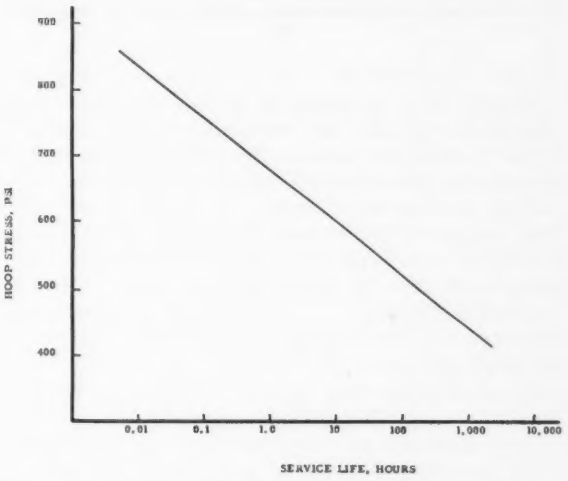

Figure 4—Hoop stress versus service life at 113 F of polyethylene pipe (annealed specific gravity 0.914).

graph enclose the experimental results and indicate the trend shown by the data.

Figure 3 shows a replotting of the average results for several series of such long-term stress tests. A line has been drawn through the lower edge of this pattern of results and extended to give an extrapolation to longer life periods. On the basis of this extrapolation, it is predicted that at 1550 psi fiber stress the acrylonitrile copolymer blend pipe would have a minimum average life of one year; at 1370 psi fiber stress, the minimum average life should be five years; and at 1330 psi fiber stress, the minimum average life should be ten years. On the basis of such data plus available field experience, the "long-term effective tensile" for the acrylonitrile copolymer blend material now used in the production of pipe has been set at 1220 psi at 73 F.

Bragaw[1] has published results of a similar study on polyethylene. Figure 4 illustrates the data for a standard high pressure polyethylene of the type used for pipe production.

Atkinson[2] quotes similar data based on German experience on unplasticized PVC. Figure 5 shows the nature of the results obtained in this work.

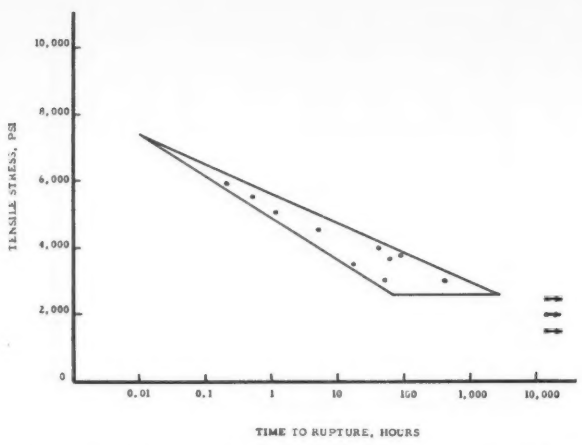

Figure 5—Tensile stress versus time to rupture at 68 F for unplasticized PVC.

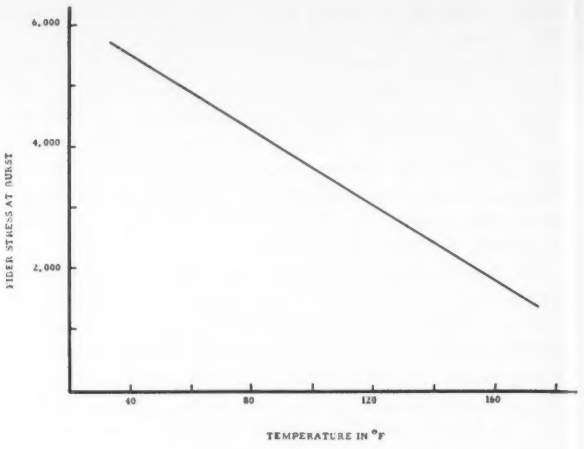

Figure 6—Fiber stress at burst versus temperature for acrylonitrile copolymer blend pipe.

Similar data on cellulose acetate butyrate, polyvinylidene chloride or American PVC Type I or Type II have not, apparently, been published up to the present time. Several papers are available indicating the long-term changes in diameter for cellulose acetate butyrate pipe at various stress levels. One paper on Type I and Type II PVC correlates creep with time by using a term called "the time dependent modulus." The data in these papers does not seem to correlate directly with the above-reported studies.

It is obvious that proven "long-term effective tensile" figures for various materials are not available at the present time. The best that can be done is to extrapolate presently available short-term and intermediate term data and correlate this in so far as possible with experience. On the basis of this, a Sub-Committee of the Thermoplastic Pipe Division of the SPI have recommended that for use *at 100 F*, the long-term effective tensiles of Type I PVC's be set at 1200 psi; the acrylonitrile copolymer blend materials and Type II PVC are rated at 1000 psi; cellulose acetate butyrate is rated at 700 psi.

Efforts to determine maximum tolerable stress levels in plastics for continuous service in pipe and/or in other shapes are not limited to this country. Technical men in Europe, working through the International Standards organization, are making estimates of values for maximum allowable stress. A recent article in *Modern Plastics*[3] quotes figures as follows:

> For rigid PVC (European material, probably similar to Type I here) 60 Kg/sq cm at 20 C (Equivalent to 850 lb/sq in at 68 F). For high pressure (low density) polyethylene 30 Kg/sq cm at 20 C (425 lb/sq in at 68 F).
>
> For rigid polyethylene (the low pressure, high density type probably made by the Zeigler method) 50 Kg/sq cm at 20 C (720 lb/sq in at 68 F).

Because of differences in temperature and even in materials tested, it is impossible at the present time to say how these figures correlate with data gathered here.

Temperature Effects

Since the materials under discussion are all thermoplastic and, therefore, soften with increasing temperature it is obvious that temperature must have a significant influence on strength characteristics. Published data indicates the effect of temperature on short-term tensile. Figure 6[4] shows the effect of temperature on the fiber stress at burst of acrylonitrile copolymer blend pipe. Figure 7[5] shows the effect of temperature on the tensile strength on Type I and Type II rigid PVC compounds. Figure 8[6] is a little bit different in that it shows the effect of temperature on the "safe hoop stress" for cellulose acetate butyrate pipe on the basis of long-term creep tests.

Another method of evaluating the strength of plastic material at elevated temperatures is the standard ASTM heat distortion test D648-45T. This test is a combination of flexural modulus and creep and therefore gives a somewhat different rating than, for example, simple tensile tests. The heat distortion temperatures for the thermoplastic pipe materials are shown in Table 2.

On the basis of such figures together with "case histories" of actual installations, the upper limits for the use of various pipes have been published in many places. Table 3 lists such recommended upper temperature limits. In the first column is indicated the range of recommendations and in the second column what seems to be the most generally used upper temperature limit.

Available Pipe Sizes

In answer to the question of what sizes of plastic pipe are currently available, a frequent answer would be "too many." Attempts are, however, being made

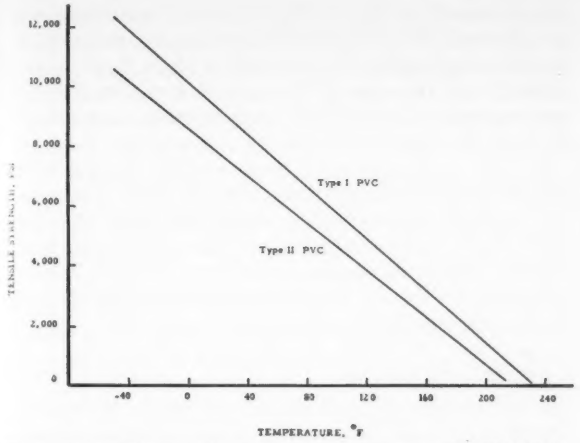

Figure 7—Tensile strength versus temperature for polyvinylchloride.

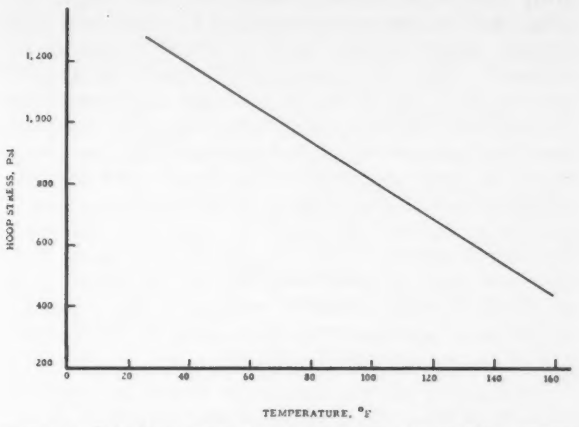

Figure 8—Safe hoop stress versus temperature for cellulose acetate butyrate pipe. Data are based on long term creep tests.

to standardize sizes. Most of the rigid pipes are being produced to IPS standards for some or all of the following schedules: 20, 40, 80 and 120. In addition to this, the SWP schedule of sizes is being used for cellulose acetate butyrate, acrylonitrile copolymer blends, and PVC. Special sizes corresponding to Type K copper tubing specifications have been proposed for insert gas services made with several of the rigid plastics. Other special sizes are being used in some applications.

Commercial standard CS197-54 has been established to specify dimensions and tolerances for flexible standard - wall polyethlene pipe. Industry standards also have been established or are in preparation for other sizes of polyethlene pipe, for cellulose acetate butyrate pipe (SWP specifications), for rigid PVC pipe, and for styrene-rubber pipe (including the acrylonitrile copolymer blend materials).

Applications of Plastic Pipe

In considering the applications of the various thermoplastics in the field of plastic piping, it is necessary to differentiate between the different types taking into account their general physical properties and chemical resistance characteristics. The special features of the various materials also will be considered.

Polyethylene

Polyethlene, because of its lower modulus, is normally classified separately from the other thermoplastic pipe materials since it gives a "flexible" pipe rather than a truly rigid one. Currently, polyethylene is used in the largest volume of any plastic pipe material. It has the lowest gravity and the lowest per pound cost; hence it may be expected to be the least expensive which will tend to maintain high volume. Its flexible nature and its toughness over a wide temperature range permit exposure to freezing without damage. Polyethylene in sizes up to 2 inches currently is supplied in long coil lengths, thus permitting economies in installation of long lines through reduction in the number of joints required and the time necessary for laying the pipe. The relatively low tensile and low softening point of polyethylene limits its use to moderate pressures and temperatures. The softness makes it susceptible to cutting damage in handling or in burial. Polyethylene has excellent corrosion resistance to practically all acids, alkalies and salt solutions except for strong oxidizing media. It is, however, susceptible to a phenomenon of stress cracking which is accentuated by certain polyoxyethylene type organic materials.

The main applications of polyethylene are in handling cold water. It is used in a large percent of jet-well installations and in many rural domestic, and farm water systems. Lawn sprinkler systems and large irrigation systems are using large amounts of polyethylene and it has found favor in handling corrosive waters in applications such as mine drainage. Its good low temperature properties have permitted its use as cooling coils in skating rinks, and it also has been used as the heat coils in radiant heating installations wherein it gains support by being buried in concrete.

Acrylonitrile Copolymer Blends

The acrylonitrile copolymer blends were the first commercial products to offer the pipe extruder a

TABLE 2—Heat Distortion Temperatures (ASTM D 648-45T)

MATERIAL	66 psi	264 psi
Polyethylene	120	105–121
Acrylonitrile Copolymer Blends	203	185–190
Polyvinyl Chloride Type I	....	160–165
Polyvinyl Chloride Type II	....	150–155
Cellulose Acetate Butyrate	171–176	146–152
Polyvinylidene Chloride	160	130–150

TABLE 3—Upper Temperature Limits

MATERIAL	Range of Recommendations	Usually Used Upper Limit
Polyethylene	105-130F	120F
Acrylonitrile Copolymer Blends	150-180F	170F
Polyvinyl Chloride Type I	130-170F	145F
Polyvinyl Chloride Type II	130-170F	140F
Cellulose Acetate Butyrate	140-160F	140F
Polyvinylidene Chloride	100-170F	....

truly hard rigid material that was also tough. Besides their unique combination of hardness and high impact, these plastics have excellent chemical resistance; they are considerably lower in specific gravity than any of the other rigid pipe materials. In addition they have high heat distortion temperature and so retain usable strength at higher temperatures. In plant operations it has been found that the materials can be reprocessed without deterioration of properties unless subjected to extreme conditions. The acrylonitrile copolymer blend compounds thus offer the pipe extruder and the user of plastic pipe an unusual combination of properties which has resulted in a continued rapid increase in the volume use of these materials for piping.

To indicate the use pattern, a recent survey indicates that after eight years of use installations are as follows: In the oil fields there is nearly 1,600 miles of lines divided almost evenly between salt water disposal and oil gathering or flow lines. For the transmission of natural gas, chiefly for the operation of irrigation pump engines, more than 2,200 miles of pipe have been installed. In industrial plants (chemical, metallurgical, and allied industries) it is found that the total is more than 530 miles, and for miscellaneous uses (potable water, underground conduit, domestic gas, and the transportation of irrigation water) the use totals more than 160 miles. This adds up to more than 4,400 miles of total pipe.

Cellulose Acetate Butyrate

Cellulose acetate butyrate gives what might really be called a semi-rigid pipe. It is intermediate in the field of non-flexible pipe materials in weight or density and has moderate chemical resistance (material is decomposed by strong acids and alkalies). Although usually pigmented black it can be produced as a clear transparent pipe, and this has opened certain special uses. It has generally good aging characteristics, although even at moderate pressures and temperature the pipe tends to creep or increase in diameter. Cellulose acetate butyrate has been used extensively in the oil and natural gas fields. It has been particularly useful in handling high paraffin crudes where it shows less tendency toward paraffin buildup.

Polyvinyl Chloride

There are two main classes of rigid polyvinyl chloride pipe compounds currently in use—Type I "normal impact" and Type II "high impact". These materials are the latest addition in this country to the field of rigid pipe manufacture, although materials of the normal impact type have been used in Europe for almost twenty years. Polyvinyl chloride has high density (relative to the other plastics), high strength, generally excellent chemical resistance, and good aging characteristics. It is self-extinguishing. Because of thermal instability at temperatures slightly above normal processing conditions, the material offers problems in production of pipe and fittings.

The Type I material may be relatively brittle and under some conditions, particularly if reworked material is used, its chemical and solvent resistance may be affected. The Type II, high impact material is, as its name implies, much tougher than Type I compounds but because of the added compounding ingredients has slightly decreased chemical and solvent resistance. The long history of use of polyvinyl chloride in Europe has been restricted largely to chemical plant installations. The rapidly expanding use of it in this country includes not only chemical plant installations but also extensive use in oil fields as flow and brine disposal lines, for handling natural gas, and for many applications in the handling of clean or corrosive waters.

Polyvinylidene Chloride

Polyvinylidene chloride is probably the oldest thermoplastic pipe materials in this country with respect to use. It has long been produced in heavy wall sizes for use in chemical plants or other locations where its excellent corrosion resistance outweighs the disadvantage of extreme brittleness. The material has the highest specific gravity of the plastic pipe materials and a very high coefficient of thermal expansion. It has been joined by the use of threaded fittings and also by hot gas welding. The material is non-flammable. In addition to its use as extruded pipe in the chemical industry, polyvinylidene chloride has found a considerable application as a chemical resistant lining for steel pipe.

New Developments

Several materials give promise of becoming factors in the field of plastic piping.

Low Pressure Polyethylene

The low pressure polyethylenes made by the various widely publicized new techniques will undoubtedly find some use as plastic piping. They are considerably stronger in short term tests than standard polyethylene, but it now appears that the long term effective tensile of the low pressure materials is likely to be only 1½ to 2 times that of the standard material. Low pressure polyethylene has a higher softening point than the standard polyethylene, but published comments on its heat resistance appear to be overly optimistic. In the standard ASTM heat distortion test at 264 psi the material shows the heat distortion temperature in the range of 120F, a lower figure than other rigid pipe materials. Its increased stiffness will take it out of the class of flexible pipe into a semi-rigid type so that insert fittings such as used with standard material will not be satisfactory. This poses a considerable problem in the joining of pipe made with the new material.

Irradiated Polyethylene

The exposure of polyethylene to high intensity radiation brings about a type of "cure", especially when the material is loaded with carbon black. Such "cured" material is more chemically resistant and has higher strength and less creep than standard polyethylene. Although the pipe is originally formed by

thermoplastic extrusion techniques, the irradiation in effect thermosets the material so that it will no longer melt. Irradiated polyethylene has been reported to have strengths as high as 500 psi at 300F. The high carbon black loading of the irradiated material has made it electrically conductive which again sets it apart from most plastic materials. The economics of the irradiation process, hence the eventual applicability in the field of pipe production, are still uncertain.

Fluorinated Hydrocarbons

Recent advances in the processing of tetrafluoroethylene and chlorotrifluoroethylene have aroused interest in these materials for use in pipes subject to unusual corrosive conditions. Costs are still extremely high relative to other plastic materials, hence the market will be of necessity relatively restricted.

Improved Materials

Considerable research is being expended in many laboratories on improvements in current pipe materials. Changes in the basic polyvinyl chloride polymer and methods of stabilization and internal lubrication can be expected to result in better "PVC" pipe materials. The acrylonitrile copolymer blend system is an extensive family of plastics and other members of this family will give stronger pipe materials serviceable over a wider temperature range. Modifications of current materials such as the terpolymers of acrylonitrile, butadiene, and styrene can be expected to give products with a new combination of properties which may prove valuable as raw materials for plastic pipe. The tremendous effort being put into the study of ethylene polymerization is expected to result in greatly increased knowledge of means of improving the overall performance of the various types of polyethylene.

Conclusions

The use of plastics for the production of pipe on a large scale dates back only about five years. Nevertheless, the industry has already grown to be an important factor in the over-all pipe field. Plastics are not going to supplant metal pipe, but have already been proven to be valuable supplements to metal pipe. Applications will certainly expand rapidly even on the basis of currently available materials, and new and improved materials can be expected to accelerate this expansion.

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Any discussions of this article not published above will appear in the December, 1957 issue.

A Review

Titanium, Zirconium and Tantalum For Control of Corrosion*

By WILLIAM E. LUSBY, JR.

Introduction

DURING THE last decade an unprecedented revolution has occurred in the metallurgical field. The evolution of the aviation gas turbine engine, high-performance airframes and the subsequent conversion of the Air Force to jet propelled craft have resulted in demands for new and improved metals and alloys. Coincident with this development has come the almost unbelievable advancement in the field of nuclear energy. Construction of equipment and plants to produce isotopes and energy has resulted in an entirely new concept for materials of construction and criteria for their selection. During this period, the metals industry has risen to the task with the development of high-temperature-resistant alloys and vacuum metallurgy. The industrial production of titanium and zirconium commenced and has assumed commercial prominence. Growth of the titanium industry has been expedited by the demands of aircraft design, while the use of zirconium has received stimulus from the field of nuclear energy.

The chemical industry has benefited greatly from the above developments. These two new metals had long been needed to bridge the gap between the corrosion resistance of stainless steel and tantalum.

In this period of an expanding economy, attended by unequaled industrial expansion, full production and rising labor costs, sound equipment design and low plant maintenance assume a position of prime significance in the manufacture of higher quality products at low cost. The availability to the corrosion engineer of titanium and zirconium as well as tantalum can aid materially in providing dependable equipment to fulfill this need.

It is the purpose of this paper to report on the progress made to date in utilization of these metals in the process industries and to outline the progress that may be expected in the next few years.

Tantalum

Tantalum is best known for its resistance to acid corrosion. It is completely resistant to hydrochloric, phosphoric, sulfuric, nitric and aqua regia at normal temperatures and pressures. Frequently, however, commercial phosphoric acid contains enough HF to seriously attack tantalum. If phosphoric acid is to be handled above 290 F, in-process tests should be conducted since some evidence indicates the metal is attacked at elevated temperatures. The halogens, chlorine, bromine, and iodine, in liquid or vapor do not react with the metal up to 300 F. Tantalum resists attack by all salts except those which hydrolyze to strong alkalis. Nearly all gases with the exception of fluorine and nascent hydrogen have no effect on the metal under 300 F. Nascent hydrogen in solution such as that due to corrosion of other metals will embrittle tantalum. Excellent resistance is shown toward most organic compounds with the exception of those that contain fluorine, free sulfur trioxide or strong alkalis.[1,2,3]

Poor Resistance to Alkalis

The Achilles heel of tantalum is its poor resistance

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Abstract

The corrosion resistant properties of tantalum, zirconium and titanium are reviewed and progress in process applications presented. Fabricating know-how and industrial experience have progressed to the point that these metals can be accepted as reliable materials of construction. Additional fundamental research is needed, however, to understand the mechanisms of attack and passivity noted. The use of these metals can now be economically justified in many locations. Volume production and lower prices forecast for titanium and zirconium due to defense needs will lead to expanded use in corrosion resistant applications. **6.5**

★ Submitted for publication June 4, 1956. A paper presented at a meeting of the Niagara Frontier Section, National Association of Corrosion Engineers, Buffalo, New York, May 10, 1956.

to alkalis or those chemicals which are strongly alkaline in nature. Its resistance to sulfuric acid breaks down at temperatures in excess of 340 F. Fuming sulfuric acid containing free SO_3 attacks tantalum even at room temperature. Hydrofluoric acid in trace amounts attacks tantalum as it does all other structural metals. A brief summary of its corrosion resistance is shown in Table 1.

Tantalum's resistance to acids and its high heat transfer coefficient have led to its widespread application in acid heating and cooling systems. A wide variety of equipment is available for this including bayonet, coil, hairpin and shell and tube heat exchangers. In most instances, these units are designed to operate at steam pressures up to 150 psi. Probably the most widespread use of tantalum in acid heaters is in Mantius concentrators for recovery of sulfuric acid. A considerable amount of the metal also is used in the construction of hydrochloric acid absorption systems.

Even in some applications where the rate of corrosion of less noble metals is sufficiently low to allow sound design, tantalum is employed to prevent contamination of the products. Process equipment for the manufacture of some pharmaceuticals and fine chemicals is a notable example of this design philosophy.

A unique application which has become significant is tantalum's use as a patch material to cover damaged areas in glass lined equipment. The metal is held in place by a stud tapped into the body of the vessel. A thin Teflon gasket makes the seal between the tantalum and the glass.

The density of tantalum is approximately twice that of steel (.6 pounds/cubic inch), and although it has a high melting point (5425 F) it reacts with gases of the atmosphere at 500-600 F. At higher temperatures it reacts with most of the elements.

High Cost

Due to the high cost of the metal ($51/lb. for sheet), tantalum usually is employed as a lining for vessels. The average thickness of liners is only .013 inch making fabrication costs greater than that of heavier gage linings. Fortunately, tantalum work hardens very slowly and reductions in thickness of up to 60 percent are possible without annealing.

Zirconium

Zirconium is next in order of corrosion resistance of the three metals under discussion. Currently, three basic types of zirconium and its alloys are produced.[4] Regular commercial grade zirconium which contains an average of 2.5 percent hafnium is usually employed for corrosion resistant applications. Zirconium from which essentially all hafnium has been removed (.02 percent hafnium max.) is referred to as Reactor Grade and finds major use in certain types of nuclear power reactors. The strength of this grade can be raised by alloying with 1.5 percent tin plus .12 percent iron, .10 percent chromium and .05 percent nickel with the resultant alloy referred to as Zircaloy II.[5]

The rapid industrialization of zirconium during recent years has been promoted by nuclear application.[4,5,6] The low thermal neutron capture of hafnium free zirconium (0.18 Barns) coupled with good strength at intermediate temperatures and excellent corrosion resistance to heat transfer media such as water or fused sodium at temperatures over 500 F and at pressures in the range of 2000 psi have led to its use in power reactor components, fuel element cladding and internal reactor structures.

The application of zirconium in the process industries is still in its infancy and limited to cases where extremely severe conditions are encountered. Laboratory investigations indicate excellent resistance to alkalis in all concentrations up to their boiling point. It is even inert toward fused caustic.[4,7] This property is in direct contrast to the poor resistance shown by tantalum.

Resistance to Acid Corrosion

The acid corrosion resistance of zirconium is somewhat better than titanium and approaches that of tantalum.[4,8,9,10] It resists attack by hydrochloric and nitric acids at temperatures up to 210 F. Resistance to mixed acid (H_2SO_4 and HNO_3) is good up to 10 percent H_2SO_4 and 90 percent HNO_3 at 210 F. Further increase in sulfuric content results in rapid attack. Its performance in sulfuric acid is more restricted, being limited to 70 percent concentration at temperatures up to 210 F. In aqua regia it behaves erratically and is not as resistant as titanium.

Experiments in hydrochloric acid show that arc-melted metal of low carbon content is fully resistant but metal containing as little as 0.1 percent carbon, such as that prepared by induction melting, becomes embrittled.[11]

TABLE 1—Corrosion Resistance of Tantalum to Various Chemicals

Generally Good Corrosion Resistance	Generally Poor Corrosion Resistance
Hydrochloric Acid	Hydrofluoric Acid
Nitric Acid	Alkalis
Sulfuric Acid[1]	Fuming Sulfuric Acid
Halogens[2]	Fluorides
Hydrogen Peroxide	Alkaline Hypochlorites
Inorganic Chlorides	
Phosphoric Acid	
Organic Acids	
Chromic Acid	
Wet and Dry Chlorine	

[1] Does not apply to fuming sulfuric acid.
[2] Does not apply to fluorides

TABLE 2—Corrosion Resistance of Commercial Grade Zirconium to Various Chemicals

Generally Good Corrosion Resistance	Generally Poor Corrosion Resistance
Phosphoric Acid[1]	Phosphoric Acid above 75 percent
Hydrochloric Acid	Moist Chlorine
Nitric Acid	Aqua Regia
Sulfuric Acid[2]	Sulfuric Acid above 70 percent
Mixed Acid[3]	Mixed Acid over 10 percent H_2SO_4
Inorganic Chlorides[4]	Ferric and Cupric Chlorides
Chlorinated Hydrocarbons	Hydrofluoric Acid
Sodium Hydroxide	Fluorides
Organic Acids	

[1] Does not apply to phosphoric acid above 75 percent
[2] Does not apply to sulfuric acid above 70 percent
[3] Does not apply to mixed acid over 10 percent, H_2SO_4
[4] Does not apply to ferric acid and cupric chlorides.

Resistance to Inorganic Chlorides

Behavior to inorganic chloride environments is excellent with the exception of ferric and cupric chlorides. Even in concentrations of 5 percent, severe attack of an embrittling nature has been noted at 210 F.[9] Resistance to chlorinated organic compounds is generally good.[10,12] The corrosion resistance of zirconium to many environments is shown in Table 2.

Above 900 F zirconium reacts with gases and at higher temperatures with all elements except the inert gases.

Conventional mill forms presently are available including sheet, plate, tubes and wire. The present price range is $20-$30 per pound for these forms. Consequently, zirconium will be applied as a lining material only for large items of process equipment for some time to come. Small valves, heat exchangers, diaphragms, and other equipment items have been fabricated. Shops equipped to handle titanium are capable of fabricating zirconium inasmuch as the two metals behave similarly in forming and welding.

Titanium

Titanium metal, which has risen to prominence in aircraft application because of its favorable strength-weight ratio, is currently enjoying expanded use in corrosion resistant applications within the process industries. During the past two years considerable effort has been devoted toward introduction of this metal into food, chemical, pharmaceutical, marine and associated fields. Although defense uses for titanium presently support the industry, it is hoped that its industrial application will be the mainstay at maturity.

The corrosion resistance of titanium is in most respects similar to that of stainless steel with, of course, several notable exceptions. Titanium cannot rival tantalum in resistance to acid environments.[8,9,13] The only acid to which it is fully resistant is nitric (except in the red fuming condition.)[14] It is readily attacked by hydrochloric and sulfuric acids at even moderate concentrations and temperatures. Phosphoric and formic acids attack the metal at elevated temperatures and concentrations.

Hydrochloric and Sulfuric Acid Service

Despite this poor resistance to acidic environments, a considerable percentage of present industrial applications is in hydrochloric and sulfuric acid service. This is due to the fact that the protective passive film on the metal can be preserved by the presence of oxidizing agents or certain metal ions in these acids.[15,16,17,18,19] The excellent resistance of titanium to aqua regia is a classic example of attack inhibited by an oxidizing agent. The addition of nitric to sulfuric acid also results in good resistance and several plant applications now take advantage of this phenomenon. Cupric, nickel, ferric, stannic and many other metal ions in low concentration have been found to inhibit the attack in acid. In general, the more noble metals are most effective.

One of the most outstanding features of titanium in regard to corrosion is its resistance to environments containing inorganic chlorides.[9] For example 8-year exposure to sea water indicates a corrosion rate of one mil per 1000 years. Titanium does not suffer pitting attack as does stainless steel in such environments, nor is it susceptible to stress corrosion cracking or intergranular attack of the heat affected zone of welds. The only reagent found to promote stress corrosion cracking is red fuming nitric acid.[20]

Chlorinated hydrocarbons and many other organic compounds do not affect titanium; however, dilute oxalic acid, trichloroacetic and citric acids at elevated temperatures attack the metal.[12] The resistance of titanium to some of the more common environments encountered is shown in Table 3.

Progress in the manufacture of mill forms has been rapid. At the present time a variety of medium strength alloys and unalloyed grades are available in most of the forms, shapes and sizes in which stainless steel is supplied. It should be pointed out, however, that the alloys now manufactured were developed as aircraft materials and may not possess the optimum properties desired for corrosion resistant service. Actually, the meager corrosion tests performed on these materials indicate resistance similar to the unalloyed metal. Since unalloyed titanium can be obtained at strength levels ranging from 40-70,000 minimum yield, it is satisfactory for most process applications.

Fabrication of Titanium

In general, titanium can be formed and fabricated by any shop proficient in fabrication of stainless steel after special instruction has been given. Steps should be taken to: (1) allow more generous bend radii than normally used for stainless steel, (2) warm-form at 5-800 F for the more severe operations, (3) fusion weld with inert gas protection and always machine with sharp tools taking generous cuts. The same care exercised for titanium must be followed in the fabrication of zirconium.

There are, however, several missing links in the fabrication practices for titanium which should be pointed out. Practical techniques are not available for producing hard surface cases needed to prevent

TABLE 3—Corrosion Resistance of Commercially Pure Titanium to Various Chemicals

Generally Good Corrosion Resistance	Generally Poor Corrosion Resistance
Nitric Acid[1]	Red Fuming Nitric Acid
Chromic Acid	Hydrofluoric Acid
Dilute Hydrochloric Acid[2]	Hydrochloric Acid above 3 percent
Dilute Sulfuric Acid	Sulfuric Acid above 4 percent
Formic Acid[3]	Formic Acid, Boiling
Lactic Acid	Phosphoric Acid
Acetic Acid[4]	Trichloracetic Acid
Inorganic Chlorides[5]	Aluminum Chloride above 10 percent
Hypochlorites	Fluorides
Moist Chlorine	Oxalic Acid
Molten Sulfur	Bromine
Chlorinated Hydrocarbons	
Chlorine Dioxide	
Food Products	
Aqua Regia	

[1] Not applicable to red fuming nitric acid.
[2] Not applicable to hydrochloric acid above 3 percent
[3] Not applicable to boiling formic acid
[4] Not applicable to trichloracetic acid
[5] Not applicable to aluminum chloride above 10 percent

galling to other metals under sliding contact nor are cast titanium parts readily available in quantity. Much work is necessary before the surface hardening problem can be overcome. Still, there is reason for optimism concerning development of titanium castings within the next two years. Recent advances in furnace technology and molding compounds are encouraging.

One of the largest areas of the use of titanium in industrial applications is in the handling of nitric acid, particularly in processes for the nitration of organic compounds. One striking example is a vertical shell and tube condenser handling 60 percent nitric acid at 480 F and 300 psi pressure. Life of the stainless steel tubes was limited to an average of six months because of severe corrosion erosion of the tubes within 4-5 inches of the inlet end. An insert of titanium was fabricated by welding 70 tubes five inches in length to a thin tube sheet cover. When in place, the titanium tubes slipped within the stainless tubes and protected the troublesome area. This unit has now been in operation for almost two years with no evidence of attack.

An interesting example of resistance to hydrochlorites is the use of titanium for the internal assembly of a filter press in which calcium hypochlorite is filtered from a slurry and compressed into solid cakes of low moisture content. Type 316 stainless steel components suffered severe pitting corrosion after two weeks of operation. The titanium parts displayed no visible evidence of attack after seven months operation. Preliminary exposure of titatium to the new chlorine dioxide bleach used in the pulp industry has shown no corrosion. This is in contrast to the poor resistance exhibited by the stainless steels and special purpose alloys in such service.

A large chlorine dioxide mixer has been lined with ⅛ inch sheet and is now in service. It is still too early to report on performance of this unit but the results obtained will indicate whether titanium will become a major material of construction in the paper industry.

Dilute Hydrochloric Acid

Although titanium does not withstand attack by hydrochloric acid in moderate concentrations, there are several production applications handling dilute acid. The first and probably the pioneer industrial application of titanium is in steam jet diffusers handling high pressure, high velocity steam, dilute hydrochloric acid and some entrained solids. The titanium diffusers have been in continuous service for more than two and one-half years without evidence of corrosion. A variety of corrosion resistant alloys were evaluated prior to the use of titanium with the best giving an average service life of three months.

In another application involving exposure to mixed organic chlorides, free chlorine and 3.5 percent HCl, titanium has performed satisfactorily for one year. Other materials failed rapidly by pitting attack when free chlorine additions were made. The unique resistance of titanium to both dilute hydrochloric acid and moist chlorine answered the need in this particular case.

The applications just discussed are but a few of many in which titanium at present prices (avg. $12.00/lb) can be justified because of savings in maintenance, downtime and lost production. Actually, the mill product cost of titanium is not a clear indication of cost to the user of process equipment since its low density and reasonable fabrication charges result in finished equipment cost much nearer that of stainless steel. With .56 pound of titanium doing the job of one pound of stainless steel on a gage for gage basis and equivalent fabrication charges, the present cost of titanium equipment is approximately three times that of stainless steel. Further reductions in mill product prices will result in more favorable equipment costs and an increasing number of industrial applications.

Summary

Of the three metals discussed, tantalum is expected to experience the least growth in corrosion resistant applications during the next decade and may, in fact, lose some volume to zirconium, particularly in hydrochloric acid environments.

Titanium will enjoy the largest growth because of volume production and, in turn, price reductions and know-how stemming from the large defense requirements. In many of its industrial applications, titanium will replace stainless steel although the expanding use of stainless steel will more than compensate for these inroads.

Zirconium will occupy the middleground. It is very nearly competitive in finished equipment form with tantalum at present and as volume rises from application in nuclear power reactors, prices should decrease.

Fundamental data on mechanisms of corrosion such as those developed for stainless steel are very limited for these metals and must be developed in order to obtain maximum utilization. Testing of titanium and zirconium in actual service applications is advancing rapidly, thus providing a wealth of practical design data in the various process industries. Equipment vendors are becoming familiar with the fabrication of titanium and zirconium as well as tantalum and are currently offering designs embodying these metals.

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TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

High Alloys to Combat Corrosion*

By E. D. WEISERT

Introduction

THE USE of highly alloyed materials to combat corrosion is associated with the more rigorous conditions encountered in industry. The applications often involve very aggressive environments, such as the hot mineral acids. In other cases, the environment may be relatively mild but product purity requirements may be such that no contamination from corrosion can be tolerated. Such applications are found in the pharmaceutical, food, and synthetic fiber industries. Use of the alloys tends to be restricted to critical areas in a process. However, such critical areas will range from minor applications, such as valve trim or stems, to major installations, including reaction vessels, pumps, piping, valves, and other equipment.

Selection of a high alloy, rather than one of the more common and less costly materials of construction, will invariably emphasize economic justification. A clear-cut justification is achieved if a high alloy is the only material showing any reasonable resistance to the environment of interest. In most cases, however, it is necessary to balance initial investment not only against anticipated life but also against maintenance costs, down time resulting in loss of salable product and safety considerations. The examples to be cited below are typical of economically justified applications.

To proceed from the selection of a material, based upon inherent corrosion resistance, to a successful application involves careful consideration of the problems of design, fabrication, and installation. Perhaps this is especially true of the high alloys. While they are amenable to ordinary methods of handling, special precautions often must be observed to ensure that the materials are exposed in a manner compatible with optimum performance. Since the investment in material is somewhat high, extra precautions are readily justified and the special characteristics of these alloys will bear discussion.

Typical analyses of the alloys to be discussed are given in Table 1. The Group 1 materials are available in either cast or wrought forms, while the Group 2 alloys are available only as castings. It is not intended that the alloys listed be considered as the only ones which may be classified as high alloys. Certainly there are other materials which might be placed into this rather loose category.

Molybdenum-Bearing Alloys

Pure molybdenum displays unusual resistance to hydrochloric and hydrofluoric acids but is rapidly attacked by strong oxidizing acids such as nitric and hot concentrated sulfuric.[1] When used as an alloying agent, molybdenum has a tendency to impart these characteristics, in some degree, to the alloy. The molybdenum-bearing 18-8 stainless steels show improved resistance to hydrochloric acid and other halogen compounds as well as to acid media in general[2]. As its molybdenum content is increased above about 22 percent, an alloy of iron containing 28 percent nickel goes from nil to very high resistance to boiling 29.8 percent sulfuric acid[3]. The addition of molybdenum to nickel imparts to the alloys corrosion-resistant properties which are very similar to molybdenum.

Nickel-Molybdenum Alloy

By taking advantage of the beneficial effects of molybdenum in a nickel base, the first wrought alloy for handling hydrochloric acid under diverse conditions was developed. Prior to 1929, equipment for use with this medium consisted of wood, rubber linings, tile, stoneware, glass, high-silicon iron, and small amounts of precious metals. Development work, described in detail elsewhere[4], led to an alloy containing 20 percent molybdenum and 20 percent iron which has good resistance to all concentrations of hydrochloric acid up to 70 C. Subsequent work[5] resulted in the 28 percent molybdenum alloy, as listed in Table 1, which is resistant to all concentrations

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Abstract

The use of highly alloyed materials to combat corrosion is discussed and extensive data are given. The following molybdenum-bearing alloys are considered at length: nickel-molybdenum alloy, nickel-molybdenum-chromium alloy, and nickel-chromium-iron-molybdenum alloy. Attention is given also to cobalt-base alloys and silicon-bearing alloys. Other matters considered in connection with these alloys include mechanical properties, welding characteristics, prewelding considerations, welding techniques, general fabrication considerations, and heat treatment. **6.5**

* Submitted for publication June 4, 1956. A paper presented at a meeting of the Niagara Frontier Section, National Association of Corrosion Engineers, Buffalo, New York, May 9 and 10, 1956.

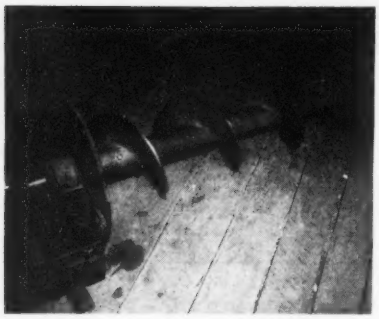


Figure 1—Cast nickel-molybdenum conveyor screw handling sulfuric acid sludge and coke.

TABLE 1—Typical Composition of Some High Alloys

Group 1-Wrought and Cast:	COMPOSITION, PERCENT									
	Mo.	Cr	Fe	Ni	Co	W	C	Si	Mn	Other
Nickel-Molybdenum	28	1 max.	5.5	65	..	..	0.08 max.	1 max.	1 max.	.....
Nickel-Molybdenum-Chromium	17	16.5	5.5	56	..	4.5	0.12 max.	1 max.	1 max.	.....
Nickel-Chromium-Iron Molybdenum	6.5	22	22	45.5	..	..	0.08 max.	1 max.	1 max.	2 Cb+T
Cobalt-Chromium Tungsten-Nickel	....	20	2	10	50	15	0.10	1 max.	1.5	.....
Cobalt-Chromium-Tungsten-Carbon	....	30	....	....	64	4.5	1.1	1 max.	1 max.	.....
Group 2-Cast Only										
Iron-Silicon	....	.....	84	....	..	....	0.85	14.5	0.65	.....
Iron-Silicon-Molybdenum	3	.....	81	....	..	....	0.85	14.5	0.65	.....
Nickel-Silicon	....	.....	....	85	..	....	0.12 max.	9.5	1	4 Cu

of the acid, even at boiling temperatures. Since it has become economically feasible to take advantage of the superior resistance of the latter alloy, the 20 percent molybdenum alloy has fallen into disuse.

The nickel-molybdenum alloy is resistant to a wide variety of non-oxidizing media, including salts, alkalis, sulfuric, phosphoric, and many organic acids. The useful resistance of the alloy to sulfuric acid is unusual among wrought materials, extending to 60 percent acid at boiling temperatures and somewhat lower temperatures for the higher concentrations. Detailed charts of the resistance of the nickel-molybdenum alloy to specific media have been reported elsewhere[6, 7, 8] and discussion here will be confined to application examples.

Nickel-molybdenum alloy is used in the production and handling of hydrochloric acid in a wide variety of equipment forms. In synthetic production, where hydrogen and chlorine gases are reacted directly, the alloy is used for reaction furnace tops where the temperature attained is about 1300 F. The product stream contains moisture, undesirable for some processes, and is brought to drying towers by alloy pipe. The drying towers contain perforated contacting trays across which the drying agent, concentrated sulfuric acid, flows. Corrosion of the first four trays is avoided by use of the nickel-molybdenum alloy. The alloy also finds application in the towers used to scrub hydrogen chloride from vinyl chloride.

To obtain an optimum surface for anodizing aluminum foil for electrolytic condensers, the foil is passed through two baths of 20 percent hydrochloric acid, one at 203 F, the other at 120 F. Heating coils, used to maintain the bath temperatures, and a series of submerged rolls, used to pass the foil through the bath, are made of the nickel-molybdenum alloy. In spite of the aeration which is present, the life expectancy of this equipment is ten years, while previous materials gave only a few months' service.

Centrifuge baskets, filter assemblies and liners are used for handling halogenated organics or inorganic halides in organic carriers in the pharmaceutical industry. Phosphorus tribromide in carbon tetrachloride and stannous chloride in alcohol are typical examples. A typical centrifuge basket may be 48 inches in diameter and 20 inches deep. Introduction of corrosive gases into the reaction mass for halogenation is accomplished with alloy piping. Pumps and other equipment of nickel-molybdenum alloy are used to handle the ethylene chlorohydrin in the production of ethylene glycol.

An outstanding usage of the nickel-molybdenum alloy has been in connection with corrosive catalysts. The alloy has unique resistance to the aluminum chloride-type catalysts as used in the Friedel-Crafts alkylation of benzene to ethyl benzene in styrene production. The isomerization of paraffinic hydrocarbons, such as butane, by use of either a hydrogen chloride-aluminum chloride-hydrocarbon complex or a melt of aluminum chloride-antimony chloride[9] is another example. The alloy also is used to handle the copper chloride catalysts in the production of chloroprene synthetic rubber. Some of the large vessels lined with nickel-molybdenum alloy for isomerization service during World War II have been successfully converted to polymerization service where phosphoric acid is the corrosive catalyst.

The conveyor screw shown in Figure 1 is of cast nickel-molybdenum alloy and is used for discharging a mixture of sulfuric acid-bearing refinery sludge and recycled coke into a roaster. The roaster produces a high-grade coke and sulfur di- and trioxides, which are converted to sulfuric acid. Portions of the screw are subjected to temperatures around 500 F. Screw conveyors of the ribbon type are used to move a titanium oxide-sulfuric acid slurry into a drying kiln. The shaft sleeve and flight, which are fabricated from plate, must withstand abrasion as well as corrosion by the slurry.

Nickel-molybdenum alloy is free from stress corrosion cracking in the boiling 42 percent magnesium chloride test[10]. Nozzles of the alloy, used to spray boiling magnesium chloride liquors into a dryer, appeared unaffected after serving for several times the life of the previously used materials.

Nickel-Molybdenum-Chromium Alloy

The ability of chromium additions to improve the passivating tendencies of iron under oxidizing conditions is well known. Chromium imparts oxidation resistance to many other metals. Broadly speaking then, chromium has the opposite effect of molyb-

denum as an alloying agent since the latter improves resistance to non-oxidizing type media. Alloys containing both elements tend to have more universal resistance as, for example, the 18-8 molybdenum stainless steel.

The development of a nickel-base, chromium-molybdenum alloy[4,5] resulted in one of the few metallic materials capable of withstanding wet chlorine gas and the alloy finds use in critical, chlorine-handling applications[11]. While resistance of the alloy to wet chlorine and hypochlorites is somewhat unique, the anticipated general resistance to both oxidizing and non-oxidizing conditions was also realized. Specific resistance of this alloy to a wide variety of media has been described in detail elsewhere[8, 12, 13]. In addition to the environments already mentioned, the alloy has good resistance to ferric and cupric chlorides and other oxidizing salts, alkalis, and organic and mineral acids.

While the resistance of the nickel-molybdenum-chromium alloy to pure sulfuric acid is not quite so good as that of the nickel-molybdenum alloy, the chromium-bearing material is more widely applied in this medium for reasons to be discussed later. This is especially true in pickling applications. In one design, pickling tanks are simultaneously heated and agitated by a submerged burner. The burner is constructed almost entirely of the nickel-molybdenum-chromium alloy. Such a burner has been in service over three years concentrating a ferric chloride solution used in tin refining. Similar burners are used to concentrate such corrosives as calcium chloride and hydrofluoric, hydrochloric, and nitric acids.

While the nickel-molybdenum alloy is usually thought of for hydrochloric acid applications, the chromium-bearing alloy has proved superior in service as a dextrose converter. The 32-foot vessel handles dextrose liquors with 0.04 normal hydrochloric acid at a pH of 1.5, operating continuously at 315-375 F and 100 psi. The corrosion rate is about four mils per year.

The chromium-bearing alloy is essentially inert in sea water and in marine atmospheres. Specimens exposed to salt water air since May, 1942 have retained their original mirror-polish finish[14]. This ability to retain a high reflectivity under corrosive conditions has led to use of the alloy as a searchlight reflector material. In ten years of immersion in sea water, no weight loss could be detected and no pitting occurred. In addition, the alloy is highly resistant to erosion by sea water at high velocities and is, therefore, useful for critical sea water service under any conditions which may be encountered. This property has been exploited in the chemical industry when sea water has been used to cool some corrosive stream.

The unusual resistance of the nickel-molybdenum-chromium alloy to wet chlorine is useful in many applications where the gas must be handled with moisture present. Injection of chlorine gas, into water systems or into chlorination reactors, is a typical application. Pumps, piping, and valves of the alloy are used to handle the chlorine in the production of synthetic glycerol and for handling chlorinated pulp stock at about 150 F in a sulfite bleach plant.

Zones of high corrosion rates have been avoided in the final towers for stripping acetic anhydride by lining the stainless steel tower with nickel-molybdenum-chromium alloy. The alloy also is being used as tower trays for some of the severely corrosive conditions in acetic acid production. Chlorides in process streams can lead to difficulties with stress corrosion cracking of the stainless steels. The chromium-bearing alloy is not affected in the boiling magnesium chloride test[10]. The alloy finds use in applications where stress corrosion cracking is a severe problem as in a carbon black slurry containing 200 ppm of chlorides.

Severe corrosive conditions exist in the first three feet of kiln-like reactors in which hydrofluoric acid is produced by the action of sulfuric acid on fluorspar. Lining this section with the nickel-molybdenum-chromium alloy has proved a satisfactory solution. In this application, the alloy must resist abrasion as well as corrosion. In another example of combined abrasion-corrosion the alloy has been used to solve a difficult pumping problem in the handling of hot phosphoric acid-gypsum slurries.

The chromium-bearing alloy also has found usage in ore leaching operations as in the processing of vanadium and uranium ores. Heat exchangers of the alloy are used to heat the sulfuric acid leaching solutions which are contaminated with a wide variety of metal ions. Spray nozzles, piping, pumps, leaf filters, and valves of the alloy are also used in connection with the ore treatments. Some application has been made in high-temperature, high-pressure processes for refining of leached ores by selective precipitation.

Nickel-Chromium-Iron-Molybdenum Alloy

Nickel-chromium-iron-molybdenum alloy, a relative newcomer to the high-alloy field[15], is similar in many ways to the nickel-molybdenum-chromium alloy just discussed. It takes advantage of the alloying effects of both chromium and molybdenum and so has a highly universal resistance, but also maintains a high iron-to-molybdenum ratio resulting in a lower-cost material. In a sense, the development work, in which 268 alloys were evaluated under six widely divergent corrosive conditions, compromised the excellent resistance of the stainless steels to strongly oxidizing conditions with the high resistance of the nickel-molybdenum alloy to non-oxidizing conditions.

One outstanding application of the alloy has developed in the pulping industry[16]. The alloy is an excellent material for use in sulfite digesters. Type 316 stainless steel lined digesters have been replacing the older brick-lined equipment, but this alloy is prone to severe pitting in the vapor phase. The high-iron, nickel-chromium-molybdenum alloy has given excellent performance as a lining in the vapor phase and liquid level areas of sulfite digesters. The strong cooking liquors used contain about 10 percent sulfur dioxide. The alloy also is useful in the new duplexing pulp process in which the first part of the cook is in either weak or strong sulfurous acid while the balance of the cook is under alkaline con-

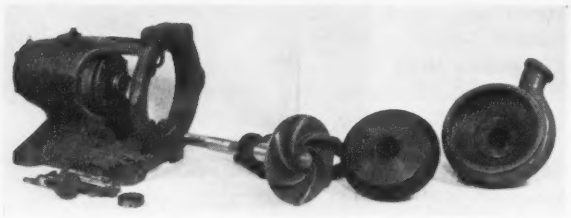


Figure 2—Portions of pump made of high-silicon iron.

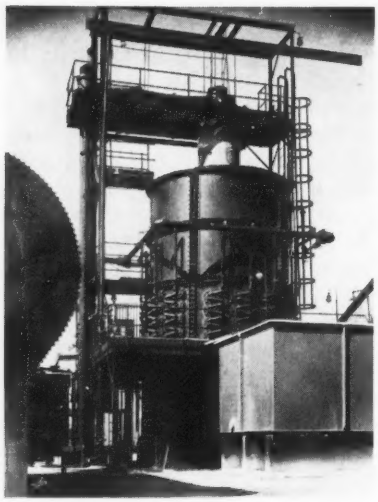


Figure 3—Simonson-Mantius sulfuric acid concentrator equipped with high-silicon nickel tubes.

ditions. The process starts at pH4 or lower and ends at about pH9. In such exposures, the alloy shows a corrosion rate of less than one mil per year in either the liquid or vapor phases.

The high-iron alloy also is completely resistant to stress corrosion cracking in the boiling 42 percent magnesium chloride test. It appears to be resistant to cracking under many of the conditions which are aggressive to austenitic stainless steels from a stress corrosion standpoint and should find application on this account. Of interest to designers of atomic reactors, this alloy has excellent resistance to supercritical water at temperatures as high as 1350 F and pressures of 5,000 psi.[17]

Cobalt-Base Alloys

Highly alloyed corrosion-resistant materials generally exhibit good strength properties at elevated temperatures. Such a dual nature is observed in stainless steels. The nickel-molybdenum and the nickel-molybdenum-chromium alloys, described earlier, have found numerous applications based primarily on their high-temperature strength. The turbine buckets of the earliest jet engines produced in this country were forged nickel-molybdenum alloy.

It is not surprising, then, that the converse also is true. In one specific case an alloy developed as a high-strength sheet material for use at 1800 F[18] has been found to be a highly corrosion-resistant material. This is the wrought cobalt-chromium-tungsten-nickel alloy.[19] The alloy has been found to have unusual resistance to hot fuming nitric acid,[20] even under erosive conditions.[21] Application experience has indicated the desirability of solution-annealing any welded structure for the more severe service conditions. Laboratory tests have shown the heat-affected zone attack to be confined to vapor phase exposure in the presence of condensation.[22]

The alloy also shows unusual resistance to wet chlorine comparable and, in certain instances, superior to the nickel-molybdenum-chromium alloy. In some applications for chlorine-handling valve stems, the cobalt material has shown up best. Though not quite comparable to the nickel-molybdenum alloy in straight hydrochloric acid resistance, there are special cases where the cobalt alloy has proven superior. An example is a 30 percent hydrochloric acid-saturated brine solution of pH 1-2 at a pressure of 60 psi and a temperature of about 220 F.

The wear-resistant and tool materials of the cobalt-chromium-tungsten-carbon type also show generally good corrosion resistance which often allows their use where ordinary wear-resistant materials fail by corrosion. Bearings and critical wear surfaces subjected to soldering flux fumes and spatter in canning machinery are typical examples. One alloy of this type has been used for years to resist the simultaneous abrasion and corrosion of machinery molding dry cell paste. Use is made of these alloys as aircraft valve trim where they must resist the erosion-corrosion of hot, sometimes lead-containing, exhaust gases. Strips of such an alloy serve as erosion shields on steam turbine blades.

Silicon-Bearing Alloys

Silicon as an alloying agent has a powerful influence on the corrosion resistance of iron-and nickel-base alloys. Silicon is known to enhance the oxidation resistance of many materials. Recent work has indicated that silicon plays a very important role in pit resistance of stainless steels[23]. Two alloys which depend upon silicon additions to achieve good corrosion resistance are the high-silicon iron, introduced in 1912, and high-silicon nickel, introduced about 1929.

Unfortunately, if the ability of silicon to impart corrosion resistance is fully exploited, unworkable alloys result and they are limited to application as castings. The castings, while of good strength, are not ductile and are subject to failure by mechanical and thermal shock. In spite of these undesirable features, the high-silicon alloys find considerable application because of their unique resistance not only to corrosion but also to abrasion and mechanical wear.

High-Silicon Irons

The high-silicon iron alloy is one of the most universally resistant alloys available. It resists almost all severe acid conditions, the notable exceptions being hydrofluoric acid, fluoride compounds, sulfurous acids, and sulfites. Improved resistance to

hydrochloric acid and chloride-bearing solutions is obtained with a modification containing 3 percent molybdenum. Data on the specific resistance of these alloys have been reported elsewhere.[24, 25]

The high-silicon irons are most widely used as pump and valve materials. Figure 2 shows pump parts made of high-silicon iron. This pump has served for more than seven years handling a pickling solution consisting of about 10 percent sulfuric acid at 160 F. A variety of other forms of equipment also is in use. By designing with the inherent brittleness in mind, it is possible to use these alloys as pipes, heating tubes, simple heat exchangers, tanks, kettles, and tower sections. The silicon-iron alloy finds wide use as chemical fume exhaust fans and as drains and traps for chemical laboratories. In the sulfuric acid field it is used in manufacture as well as handling. The good abrasion resistance of the alloy is exploited in applications such as pumping sulfuric acid-titania slurries. The alloy is used in the concentrating of sulfuric acid as heating tubes, pipes, fittings, and tank outlets. It is especially resistant to hot strong nitric acid and vapor. In the saltpeter process, high-silicon iron retorts, denitrators, towers, condensers, coolers, pipes, and valves are used.

The molybdenum modification is primarily applied for chloride and hydrochloric acid service. A valve of this alloy has successfully handled tin tetrachloride for over 10 years. In an application pumping zinc chloride flux, the alloy has given five years' service.

Nickel-Silicon-Copper Alloy

In searching for an alloy of the high-silicon iron type based on nickel, the nickel-silicon, nickel-aluminum systems were investigated.[4] Briefly, it was found that up to 10 percent silicon, the nickel-silicon alloys were strong and fairly tough but not particularly resistant to severe corrosion. At 15 percent silicon they have excellent resistance but are extremely brittle. A similar situation was found in the nickel-aluminum system, with the brittleness and good resistance occurring at about 20 percent aluminum. In the ternary system a useful alloy was found at 10 percent silicon, 5 percent aluminum. Copper was found to be a satisfactory replacement for most of the aluminum and further increased the toughness. A nominal composition of 10 percent silicon, 3 percent copper, and a small amount of aluminum was established and marketed. In later work the aluminum content was conceded to be detrimental to welding characteristics and was dropped with very little loss of corrosion resistance.

The high-silicon nickel alloy is not superior to the high-silicon iron alloy in corrosion resistance. However, in many applications it has been found advantageous to accept the somewhat lowered resistance in view of its considerably higher strength and resistance to thermal and mechanical shock. Heating tubes for the Simonson-Mantius sulfuric acid concentrator, shown in Figure 3, serve to illustrate this comparison. High-silicon iron tubes show low corrosion rates but the heating medium is limited to about 100 psi steam to avoid thermal shock failure. With high-silicon nickel tubes, 300 psi steam or Dowtherm may be used to achieve considerably faster production rates.

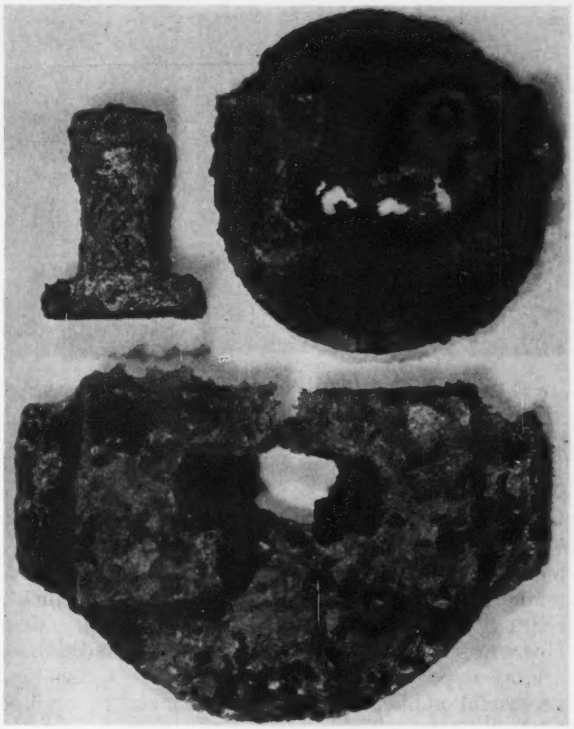


Figure 4—Nickel-molybdenum valve parts which failed when coupled with carbon.

TABLE 2—Effect of Sludge Contamination and Galvanic Coupling with Graphite and Carbon on the Resistance of Nickel-Molybdenum Alloy to Sulfuric Acid

Medium	CORROSION RATE IN BOILING MEDIUM, MILS PER YEAR	
	Cast	Wrought
ca. 50% Sulfuric Acid-Hydrocarbon Sludge, Containing SO_2	37[1]	41[1]
50% Sulfuric Acid, C.P. Grade	6[1]	4[1]
50% Sulfuric Acid, C.P. Grade Coupled with Graphite	531[2]	507[2]
50% Sulfuric Acid, C.P. Grade Coupled with Carbon	1,960[3]	1,850[3]

[1] Average rate for five 24-hour periods.
[2] Average rate for three 24-hour periods.
[3] Average rate for two 24-hour periods.

While it is used primarily for sulfuric acid applications, the high-silicon nickel alloy has good resistance to a wide variety of media, which have been reviewed elsewhere[8, 26]. Like the iron-base alloy, it has excellent abrasion resistance which is exploited in applications such as check ball valves, piston rods, shaft sleeves, and pump parts. Tubes of the alloy are used for introducing the hot air into the sulfuric acid bath in the Chemico-type concentrator. In a similar application, high-silicon nickel alloy downcomers are used to introduce hot benzene vapors into hot concentrated sulfuric acid in the production of benzene sulfonic acid as a phenol intermediate.

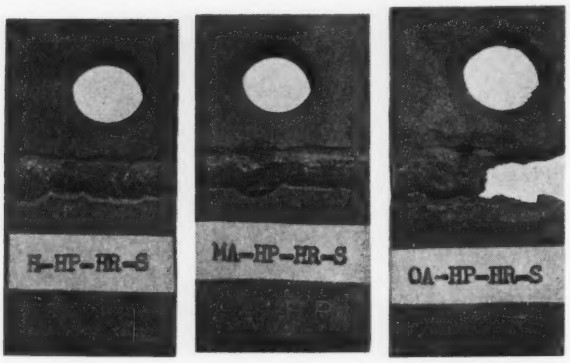


Figure 5—Effect of welding method on the corrosion resistance of nickel-molybdenum-chromium alloy.

General Considerations of Corrosion Behavior

The high-alloy materials can be used to solve many difficult corrosion problems. Their general tendency toward universal resistance is a valuable characteristic and offers wide latitude in application. However, it should be emphasized that selection of a more expensive alloy does not, in itself, assure a successful application. The alloys must be applied with good engineering discretion, keeping in mind that they are subject to the same basic principles of corrosion as are the more common materials of construction.

The excellent resistance of the wrought cobalt-chromium-tungsten-nickel alloy to various chlorine environments has been mentioned. In one case a test showed the alloy to have original polish marks after six months of exposure to a liquid hydrocarbon stream containing chlorine and water. In the same test the nickel-molybdenum-chromium alloy showed heavy etching and pitting. However, the cobalt alloy also showed severe pitting underneath retaining washers. If the good resistance of the cobalt-base alloy is to be exploited in this environment, equipment will have to be carefully designed so as to avoid any sites at which crevice corrosion could occur.

The possibilities of galvanic corrosion should not be overlooked. In general, the high alloys will tend to be the more noble member of a couple and it may be anticipated that any difficulties encountered will tend to affect the lower alloy material. Galvanic attack of the high alloys, however, is sometimes encountered. The very drastic failure of two nickel-molybdenum valves after five or six weeks' service, is shown in Figure 4. Selection of the alloy to serve in a stream consisting of about 50 percent sulfuric acid, various hydrocarbons, and some sulfur dioxide at 275 F, seemed justified on the basis of the resistance of the alloy to sulfuric acid under such conditions. Contamination of the sludge, especially with oxidizing agents, was one possible explanation of the failure but did not seem to account for the drastic nature of the attack. Erosion-corrosion was ruled out because of the severe attack on areas not exposed to high velocities. Knowledge that the valve had been installed in contact with carbon pipe liners suggested the possibility of galvanic attack.

TABLE 3—Effect of Oxidizing Cation Contamination on the Corrosion Rates of Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloys in Sulfuric and Hydrochloric Acids

Alloy	Corrosion Medium	CORROSION RATE-MILS PER YEAR (24 Hour Periods-Except Where Noted) 1st	2nd	3rd	4th	5th	6th	7th	8th
Ni-Mo	50% H_2SO_4, boiling	59	20	22	..	..	..	..	..
Ni-Mo	Same plus 0.01% Cu as $CuSO_4$	171	119	123	..	..	..	..	..
Ni-Mo	Same plus 1.0% Cu as $CuSO_4$	937	601	696	..	..	..	..	..
Ni-Mo-Cr	98% H_2SO_4 @ 200F	4	5	8	15	11[1]	22[1]	20[1]	22[1]
Ni-Mo-Cr	Same plus 25 ppm Cu as $CuSO_4$	6	6	6	6	..	..	..	..
Ni-Mo	1% HCl, boiling	6	10[2]	13[1]	..	..	..	..	..
Ni-Mo	Same plus 0.02% $FeCl_3$	23	40[2]	30[1]	..	..	..	..	..
Ni-Mo-Cr	1% HCl, boiling	35	40[2]	78[1]	..	..	..	..	..
Ni-Mo-Cr	Same plus 0.02% $FeCl_3$	8	8[2]	8[1]	..	..	..	..	..

[1] 48-hour period.
[2] 41-hour Period.

The test data presented in Table 2 show that there was an effect of contamination, largely copper, which accounted for about a tenfold increase of corrosion rate in the sludge as compared to chemically pure sulfuric acid. However, the rate of 40 mils per year is not unreasonable for severe service and does not account for the drastic attack observed. Coupling of the alloy with graphite, using the chemically pure acid, gave a hundredfold increase in corrosion rate

TABLE 4—Typical Mechanical Properties of Some High Alloys

	GROUP 1—WROUGHT and CAST									
	Ni-Mo		Ni-Mo-Cr		Ni-Cr-Fe-Mo		Co-Cr-W-Ni		Co-Cr-W-C	
	Wrought	Cast	Wrought	Cast	Wrought	Cast	Wrought	Cast	Wrought	Cast
Ultimate Tensile Strength, psi x 10^{-3}	135	80	130	78	106	73	150	112	200	120
Yield Strength (0.2% offset), psi x 10^{-3}	57	57	55	57	50	37	68	...	...	...
Elongation, Percent	52	8	45	8	52	21	55	35	5	1.5
Hardness, Rockwell B	95	94	95	98	84	83	98	90	Rc43	Rc46
Impact Strength, Notched Izod, ft-lb	60	18	70[2]	20	80	..	120+	..	..	11[1]
Modulus of Elasticity, psi x 10^{-6}	30.7	26.5	28.5	26	29	..	35.3	..	30	30

	GROUP 2—CAST ONLY		
	Ni-Si	Fe-Si	Fe-Si-Mo
Ultimate Tensile Strength, psi	117,000	16,000	16,000
Elongation, Percent	0–1		
Hardness	Rc34	520 Brin.	520 Brin.
Impact Strength, Izod, ft-lb	1–2		

[1] Unnotched
[2] Notched Charpy

TABLE 5—Typical Mechanical Properties of the Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloys in the As-Welded Condition[1]

Alloy	Weld Method	Specimen Thickness in.	Ultimate Tensile Strength, psi	Yield Strength 0.2% Offset psi	Elong. in 2 in.,%	Angle of Deformation After 180-degree Transverse Bend	Weld Failure	HARDNESS, R_B		
								Plate	By Weld	In Weld
Ni-Mo	Metallic Arc	1/8	121,600	67,500	16	166	No	R_A62	R_A61	R_A53
Ni-Mo	Metallic Arc	1/4	123,900	55,500	40	O.K.	No	91	92	93
Ni-Mo	Metallic Arc	3/8	121,100	59,600	37	O.K.	No	97	97	93
Ni-Mo	Inert-gas-shielded-arc	1/8	117,200	58,800	30	172	No	R_A57	R_A59	R_A55
Ni-Mo	Inert-gas-shielded-arc	1/4	115,000	60,100	30	O.K.	No	92	93	95
Ni-Mo	Inert-gas-shielded-arc	3/8	124,900	48,500	42	O.K.	No	92	94	90
Ni-Mo	Min. Prop. fully-annealed base alloy ASME, Case 1173	to 3/16	115,000	48,000	45	....	..	....	....	....
Ni-Mo	Min. Prop. fully-annealed base alloy ASME, Case 1173	to 3/4	100,000	45,000	40	....	..	....	....	....
Ni-Mo-Cr	Metallic Arc	1/8	116,900	68,300	19.5	167	No	R_A59	R_A60	R_A60
Ni-Mo-Cr	Metallic Arc	1/4	128,500	66,500	28	O.K.	No	87	90	91
Ni-Mo-Cr	Metallic Arc	3/8	118,000	65,900	22	O.K.	No	R_A52	R_A51	R_A54
Ni-Mo-Cr	Inert-gas-shielded-arc	1/8	121,000	66,000	25	178	No	R_A60	R_A57	R_A60
Ni-Mo-Cr	Inert-gas-shielded-arc	1/4	113,200	58,900	32	O.K.	No	97	97	96
Ni-Mo-Cr	Inert-gas-shielded-arc	3/8	116,500	64,000	25	O.K.	No	R_A54	R_A56	R_A56
Ni-Mo-Cr	Min. Prop. fully-annealed base alloy ASME, Case 1194	to 3/16	115,000	50,000	40	....	..	....	....	....
Ni-Mo-Cr	Min. Prop. fully-annealed base alloy ASME, Case 1194	to 3/4	100,000	45,000	30	....	..	....	....	....

[1] Adapted from Reference 29

over the uncoupled rate, while coupling with amorphous carbon increased the rate about 500 times. The area of the graphite and carbon was about 18 times that of the metal specimens but it was estimated that the effective ratio would be even greater under service conditions. These data seem to account for the catastrophic attack and give warning against coupling the nickel-molybdenum alloy with either carbon or graphite under similar conditions.

In the example just cited, contamination was mentioned as affecting the rate of corrosion of the nickel-molybdenum alloy. The alloy is somewhat analogous to the noble metals in that it has little tendency to displace hydrogen from acid solutions under a wide variety of conditions. The rate at which it corrodes is primarily controlled by cathodic reactions so that accelerated corrosion is incurred by depolarizing actions at the cathode areas. Thus, excessive dissolved oxygen and oxidizing cations have an accelerating effect. On the other hand, the chromium-bearing nickel-molybdenum alloys seem to derive their resistance in part from an anodic passivation tendency similar to the stainless steels. The presence of oxygen or oxidizing contaminants tends, if anything, to enhance their resistance by maintaining and repairing the passive condition. The data in Table 3 illustrate these effects. The presence of the cupric ion in sulfuric acid accelerates the corrosion rate of the nickel-molybdenum alloy but tends to inhibit the corrosion of the chromium-bearing alloy. In hydrochloric acid the nickel-molybdenum alloy corrodes more rapidly in the presence of the ferric ion while the chromium-bearing alloy is again inhibited, strongly in this case.

The silicon-bearing alloys seem to depend primarily on film formation for their resistance. They are unaffected by contamination except of a nature which might attack or increase the solubility of the scale formed on the alloy.

Mechanical and Metallurgical Characteristics

Mechanical Properties

Table 4 lists some typical mechanical properties of the alloys under discussion. They are, with exception of the high-silicon irons, high-strength materials. The wrought alloys exhibit good ductility and are amenable to ordinary cold-working and fabrication methods. Their forming behavior is very similar to the austenitic stainless steels, taking somewhat more power because of their higher strength. Forming and shearing power requirements are approximately twice those for ordinary steels. Since the alloys are face-centered cubic, they exhibit considerable work-hardening tendencies, which are used to advantage on some applications requiring wear resistance.

The nickel-molybdenum alloy and the nickel-molybdenum-chromium alloy have both been accepted into the ASME Boiler and Pressure Vessel Code so that formalized maximum stress allowances at various temperatures are available[27, 28]. As has previously been mentioned, these alloys tend to retain good strength at elevated temperatures so that strength is seldom a limiting factor in high-temperature corrosion applications.

Welding Characteristics

The highly-alloyed materials can be successfully welded by conventional methods. Electric arc welding with a coated electrode and inert-gas-shielded arc welding are the preferred and most commonly used methods for the wrought alloys. As with other highly-alloyed materials, careful welding practices must be adhered to if unsatisfactory joints and cracking are to be avoided. While the alloys are amenable to oxy-acteylene welding, this method is avoided to prevent loss of corrosion resistance due largely to carbon pickup. This is dramatically illustrated in Figure 5. These welded specimens of the nickel-

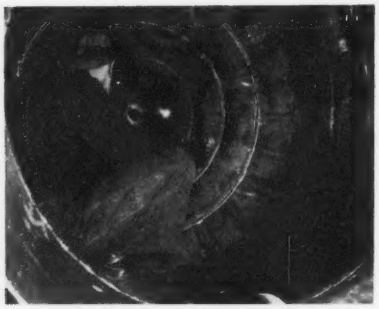


Figure 6—Section of strip-lined preheater for benzene hexachloride production.

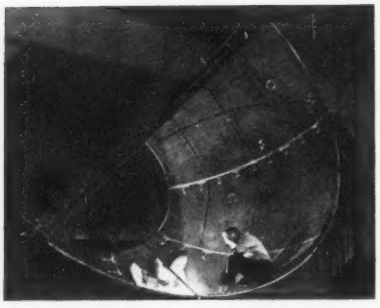


Figure 7—Head of a sulfite digester lined with nickel-chromium-iron-molybdenum alloy by spot resistance welding.

molybdenum - chromium alloy were exposed, as-welded, to conditions which attacked the weld metal more severely than the parent plate. The only difference between the specimens is the method of welding, which (from left to right) was inert-gas-shielded arc, metallic arc, and oxy-acetylene.

When good welding techniques are used, joints of very high efficiency and good ductility result as indicated by the data in Table 5. The optimum procedures for joining the wrought alloys have been discussed in detail elsewhere[29], and only the more important considerations will be abstracted here.

Prewelding Considerations

It is imperative that the surface to be welded be free of all grease, dirt, chalk or crayon markings, or other foreign matter. Joint designs are similar to those used for steel but clearances are generally larger. Backing rings are avoided in corrosive service to avoid crevices where concentration cells may form. Beveled edges are used for material heavier than 12 gage and welding is done from both sides. For the thinner material, welding may be accomplished from one side, but in all cases complete penetration is a criterion of a good weldment.

Edge preparation is best obtained by machining. Thermal cutting and beveling are possible but not recommended because of the tendency to locally sensitize the alloys over and above any effect of welding heat. Sheared edges should not be welded directly but should be ground back at least 1/16 inch or, alternately, full-annealed prior to edge preparation. The severe stress pattern residual in the sheared edge can be a strong factor contributing to welding cracks.

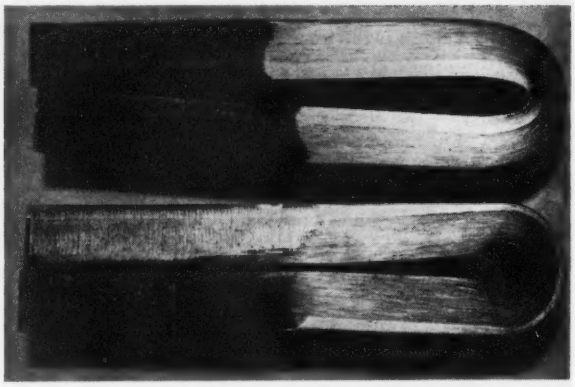


Figure 8—Bend tests of nickel-molybdenum braze-clad material.

Alignment of the prepared edges is very important since misalignment leads to variations in bead contour, gap width, and stress in the weld area, all contributing factors to cracking. The proper alignment should be retained by tack welding. Jigs and fixtures may be used to good advantage to maintain alignment and facilitate welding. Figure 6 shows a section of a strip-lined preheater for benzene hexachloride production. In this case, a special fixture positioned the vessel in a manner that all welds could be made in the downhand position.

Welding Techniques

In general, these alloys are amenable to the same welding techniques as used for the austenitic stainless steels. The following precautions are emphasized:

1. Use stringer beads at all times (no weaving)
2. Do not preheat or postheat
3. Keep weldment as cool as possible, using quenching techniques where practical
4. Use striking tabs where possible

These precautions are primarily aimed at keeping the heat content of the metal at a minimum since cracking is asociated with a lowered ductility at intermediate temperatures which compounds the cracking tendencies due to thermal stress alone.

Cracking rarely occurs in the weld bead. The most commonly encountered cracking is in the parent metal adjacent and transverse to the weld bead. If the design is such that the materials are held under restraint during welding, cracking of the parent metal parallel to the weld bead may be encountered. An obvious situation where this might occur is during strip lining of a vesel. After welding three sides of a segment, it is under restraint and cracking might be encountered while welding the fourth side. In one case, during the lining of large isomerization vessels, a crimp was put in each segment prior to welding, which served to alleviate the restraining stresses.

Electrode coatings developed for these alloys allow deposition of porosity-free beads. When using the inert-gas-shielded arc method, porosity may be encountered if the shielding gas is dispersed, as by drafts, or if the nitrogen content of the gas is too high.

The high-silicon cast alloys can be welded by the oxy-acetylene method.[8, 24] Preheating is required to 1600 F for the iron-base alloy and to 1200 F for the nickel-base alloy. Slow cooling after welding is recommended. For the high-silicon nickel an excess acetylene flame is recommended. Special welding fluxes are sometimes used. For the iron-base alloy equal parts of lime and sodium bisulfite are used. The molybdenum-bearing, high-silicon iron is considerably more difficult to weld than the parent alloy, and the manufacturer's advice on procedures should be solicited.

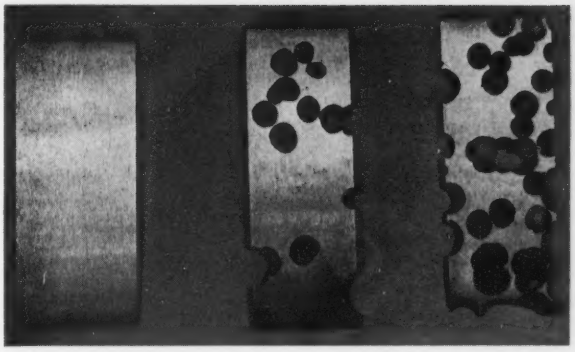
General Fabrication Considerations

Machining behavior of the molybdenum-bearing alloys is again similar to the austenitic stainless steels, but somewhat more difficult due to their higher rate of work-hardening. Because of this tendency, it is important to use sharp tools, to keep them engaged as continuously as possible, and to have a firm, rigid, work setup. Detailed machining and grinding recommendations are available.[8]

Scale removal after heat-treatment is most readily accomplished for the wrought alloys with a molten caustic or molten sodium hydride bath.(1) The alloys are water-quenched on removal from the molten bath and dipped in an acid bath for final cleaning. For the nickel-molybdenum alloy a 12 percent sulfuric acid at 135 F is used while a 10 percent nitric acid, 1-2 percent hydrofluoric acid at 175 F is used for the chromium-bearing alloys.

When large vessels are to be fabricated, construction from solid alloy material, while feasible from a strength, design, and fabricability standpoint, may be difficult to justify economically. Since clad plate is only recently available, strip lining as shown in Figure 6, has been the most widely used method to apply the alloys in large vessels. An alternate means is a proprietary process(2) which achieves the lining by spot resistance welding on close centers. The head of a large sulfite digester, lined with the nickel-chromium-iron-molybdenum alloy in this manner is shown, during fabrication, in Figure 7.

A recent innovation in the cladding field[30] may prove to be a successful method of producing clad plate of the wrought alloys under discussion. The method(3) depends upon a high-temperature braze to bond alloy sheet of the required thickness and an appropriate constructional steel to obtain a clad material without rolling. A sandwich is prepared of steel-brazing material-alloy-alloy-brazing material-steel. This assembly is edge welded and a vacuum applied internally so that the pack is compressed by atmospheric pressure. The whole assembly is then brought to brazing temperatures in a furnace. After cooling, the edge welds are removed to separate the two clad plates. Bend tests performed on nickel-molybdenum alloy clad in this manner are shown in Figure 8. While still in a development stage, with respect to the alloys under discussion, this method of producing clad material of desirable properties looks very promising. An even more recent development(4) has resulted in the successful roll cladding of several of the wrought alloys. Thus the availability of clad materials as a standard product appears imminent.



Annealed Sensitized Sensitized

Figure 9—Annealed and sensitized specimens exposed to boiling ferric chloride.

Heat-Treatment and Microstructure

The analogy of the Group 1 alloys to the austenitic stainless steels is extended to consideration of their response to heat-treatment. These alloys are austenitic in nature; that is, they consist of a face-centered-cubic matrix, which is a nickel-rich or cobalt-rich solid solution, plus some insoluble constituents. At sufficiently elevated temperatures, there is a tendency for a maximum amount of the alloy components to dissolve into the matrix. This solution-annealing temperature range is shown for the various alloys in Table 6. The alloys are believed to be essentially two-phase in the solution-annealed condition, consisting of the austenitic matrix and insoluble carbides of the M_6C type.

At intermediate temperatures, roughly 1200 F to 1900 F, the matrix is in a supersaturated condition and precipitation of secondary constituents can occur. In the chromium-bearing alloys, the precipitation appears to be largely the cubic chromium carbide, $Cr_{23}C_6$. Intermetallic compounds may also be encountered. Precipitation of this type in the nickel-molybdenum alloy is very sluggish, except under the influence of prior cold-work, and consists primarily of nickel-molybdenum intermetallic compounds.

The effect of precipitation at intermediate temperatures is to sensitize the alloys, making them more prone to corrosion, especially to corrosion of an intergranular nature. The degree of sensitization will

(1) Proprietary processes: caustic—Hooker Electro Chemical Company, Niagara Falls, New York. Sodium hydride—E. I. duPont de Nemours and Company, Wilmington, Delaware.

(2) Smithlining, A. O. Smith Corporation, Milwaukee, Wisconsin.

(3) Hortonclad, proprietary process of the Chicago Bridge and Iron Company, Birmingham, Alabama.

(4) Development by Lukens Steel Company, Coatesville, Pennsylvania.

Figure 10—Solution-annealed structure, nickel-molybdenum-chromium alloy.

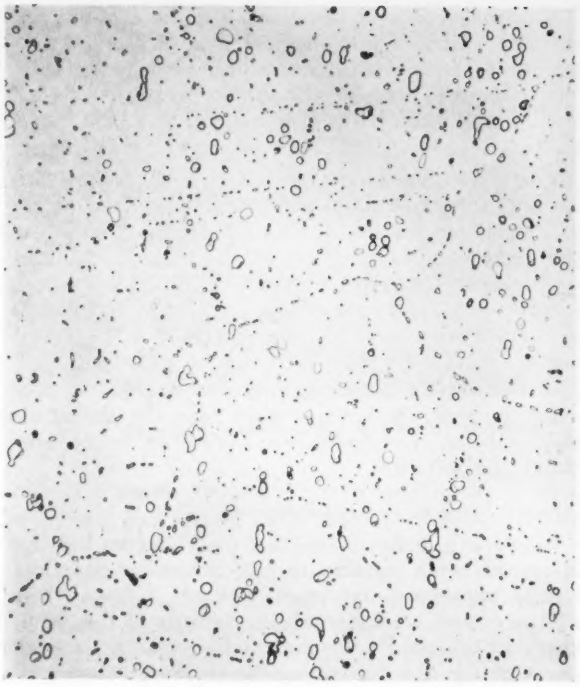


Figure 11—Structure as annealed at too low a temperature.

be a result of the temperature(s) and duration of exposure. In a very aggressive environment, the effect of sensitization can be dramatically illustrated. Samples of the nickel-molybdenum-chromium alloy were exposed for six hours to a boiling, 10 percent ferric chloride solution. The result is shown in Figure 9. The specimen on the left was in the solution-annealed condition. The two specimens on the right had been severely sensitized by two different procedures.

Optimum corrosion resistance and mechanical properties are developed by a solution-annealing treatment followed by cooling through red heat as rapidly as possible. Water-quenching and rapid air-cooling by means of a fan are preferred in that order. Cooling in still air should be avoided. While solution-annealing is recommended for all cases where it is feasible, there are many cases in which it is impossible or impractical to anneal the fabricated structure. Perhaps more often than not, the alloys are applied in the as-welded condition. In the majority of cases, this is a satisfactory procedure. However, in very severe environment preferential attack of the heat-affected zone and/or the weldment may be encountered. For this reason, application of as-welded structures in particularly arduous media is best preceded by testing of as-welded samples. The success of an as-welded structure in service will depend a great deal upon the use of good welding techniques.

Using the nickel-molybdenum-chromium alloy as an example, typical microstructures are shown in Figures 10 through 16. The solution-annealed structure, Figure 10, shows no grain boundary precipitation and consists of an unstrained matrix and insoluble carbides. If the annealing temperature is too low, the structure shown in Figure 11 results. Both grain boundary precipitation and precipitation within the grains are observed. The discontinuous, spheroidal nature and the large size of the grain boundary precipitates indicate that they were formed at temperatures lower than the nominal solution-annealing temperature for the alloy, 2230 F, by about 150 F to 300 F.

If the alloys are overheated, incipient melting will occur primarily in the grain boundaries and to a lesser extent at sites of primary carbides. A clear-cut case of incipient melting is shown in Figure 12. If the incipient melted structure is less well developed, it may easily be confused with the structure shown in Figure 13. This structure is characterized by the continuous grain boundary precipitation which results from cooling too slowly through the range of sensitization temperatures. When not obvious metallographically, the two structures may be differentiated by solution-annealing and water-quenching. The incipient melted structure will persist through such a treatment while the sensitized structure will not.

In solution-annealing any welded structure, it is advisable to allow at least one hour at the annealing temperature. This serves to homogenize the weld metal, breaking up some of the dendritic segregation. Figure 14 shows the structure of a weld of the nickel-molybdenum-chromium alloy in the as-welded condition. After partially annealing, the structure is as shown in Figure 15. In Figure 16 a fully-annealed weld structure has been achieved. Two effects are

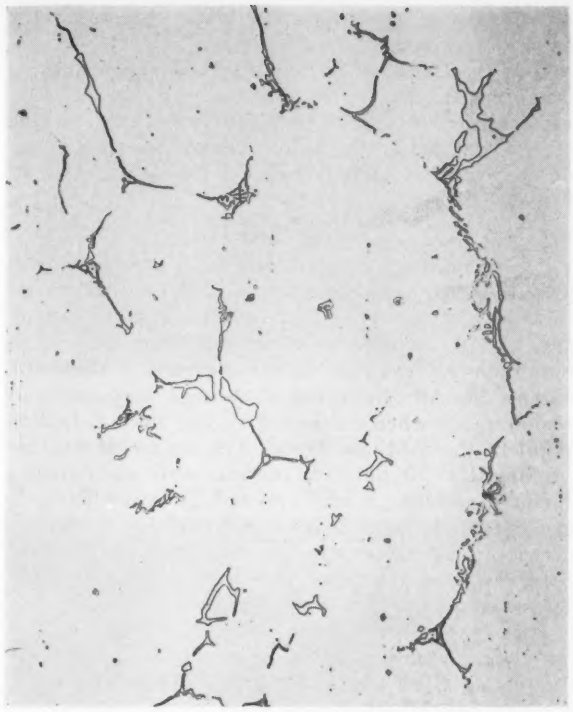


Figure 12—Structure after incipient melting has occurred.

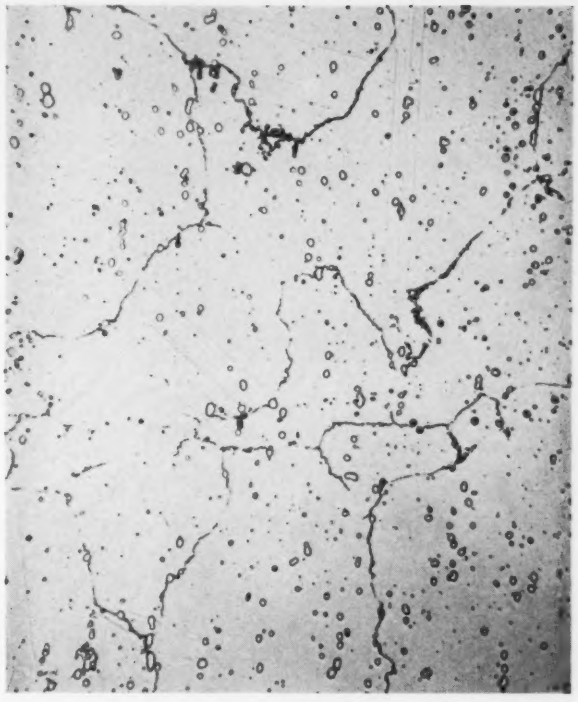


Figure 13—Structure obtained by slow cooling.

Figure 14—Structure of nickel-molybdenum-chromium alloy weld metal.

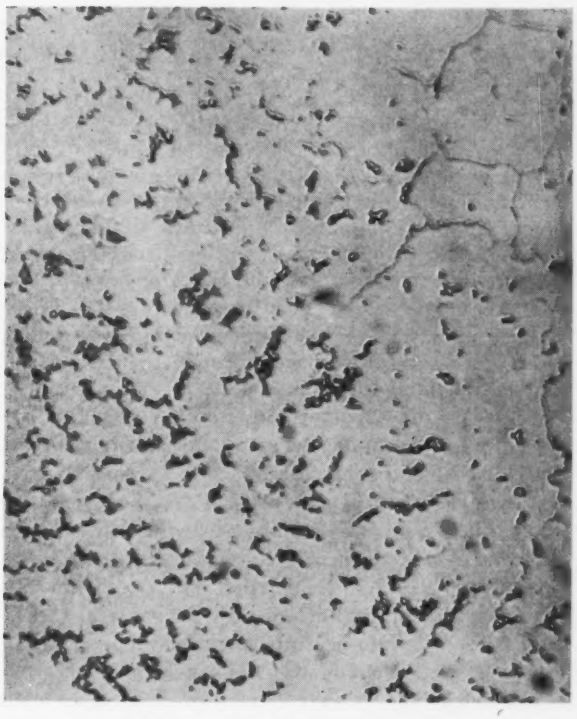


Figure 15—Structure of weld after partial annealing.

noticed: (1) some of the material observed in the as-welded condition has gone into solution, and (2) the carbides have agglomerated and spheroidized. Residual evidence of incipient grain boundary melting may be noticed adjacent to what was the molten puddle.

During the annealing of the alloys containing molybdenum, precautions should be observed to

Figure 16—Structure of weld after complete annealing.

avoid local oxidation of a rapid nature. This occurs when a surface of the workpiece is exposed to stagnant conditions which allow a concentration of molybdenum trioxide, a volatile constituent of the normal scale, to build up. A flat surface resting directly on a furnace hearth would be subject to this attack because of the stagnation in the crevice formed. Foreign matter on the workpiece may be a source of local stagnation and attack. Rapid oxidation is best avoided by assuring good circulation of the furnace atmosphere around the workpiece, which tends to carry away the volatile oxide.

Since oxidation of molybdenum-containing alloys at high temperatures is accompanied by a volatilization of the molybdenum trioxide from the scale, it was of interest to determine if this loss would be accompanied by a depletion of molybdenum in the unoxidized metal. The data in Table 7 were obtained, for the nickel-molybdenum alloy, by an X-ray fluorescence analysis technique in which the surface was analyzed after successive removals of 0.0002-inch layers. It is seen that a depletion does occur and that the apparent depth affected is approximately 0.8 mil. For the chromium-bearing, nickel-molybdenum alloy no depletion could be detected. The results obtained with this technique were in good agreement with emission spectrographic data obtained on a tapered specimen.

Heat-treatment of the high-silicon nickel alloy is primarily for purposes of stress-relieving. This is accomplished at 1800 to 1825 F followed by a furnace cool. Maximum hardness for abrasion resistance may be developed at 1100 F, soaking for two hours, and furnace cooling.

TABLE 6—Solution-Annealing Temperatures for Group 1 Alloys

Nickel-Molybdenum	2100-2150F
Nickel-Molybdenum-Chromium	2210-2160F
Nickel-Chromium-Iron-Molybdenum	2100-2150F
Cobalt-Chromium-Tungsten-Nickel	2225-2275F
Cobalt-Chromium-Tungsten-Carbon	2150-2225F

Future Trends

The trend toward higher process temperatures and pressures has stimulated corrosion studies under these conditions[31, 32] which indicate that high-pressure conditions may be very demanding on present materials. An example of these effects is shown in Figure 17. All alloys tested showed very low corrosion rates when subjected to the normal boiling point of the medium, 216 F. The corrosive was approximately 10 percent sulfuric acid containing furfural residue. When exposed, in autoclaves, to the operating conditions of 100 psi, 340 F, the increase in corrosion rate of one alloy was almost 5,000-fold and the alloy that appears worst at the lower temperatures now appears best.

It is ever the aim of the corrosion engineer to find ways of making ordinary materials perform satisfactorily under arduous conditions. These efforts are pursued in the interest of conservation as well as economic considerations. However, it is clear that these efforts will not alone suffice to satisfy the future requirements of the process industries. Processes utilizing extremely severe conditions will be found attractive and materials capable of handling these conditions will be sought. The use of high alloys may be expected to increase, and the development of new, special materials will be justified by the economic advantages of the new processes.

Acknowledgments

The author is indebted to the Development and Technical Services Department of the Haynes Stellite Company, where much of the above information was developed. Particular thanks are due to J. R. Schley and R. P. Culbertson of that group. The cooperation of W. A. Luce of The Duriron Company, with respect to the high-silicon irons, is gratefully acknowledged.

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TABLE 7—Surface Depletion of Molybdenum During Processing of Nickel-Molybdenum Alloy

Condition of Alloy[1]	Percent Molybdenum in Surface, Obtained by X-ray Spectrograph				
	Original Surface	0.0002 Inch Removed	0.0004 Inch Removed	0.0006 Inch Removed	0.0008 Inch Removed
As-Rolled	25.50	26.65	26.95	27.15	27.35
Annealed at 2150F	22.95	24.65	26.25	27.05	27.25

[1] Master Heat Analysis 27.23% Molybdenum.

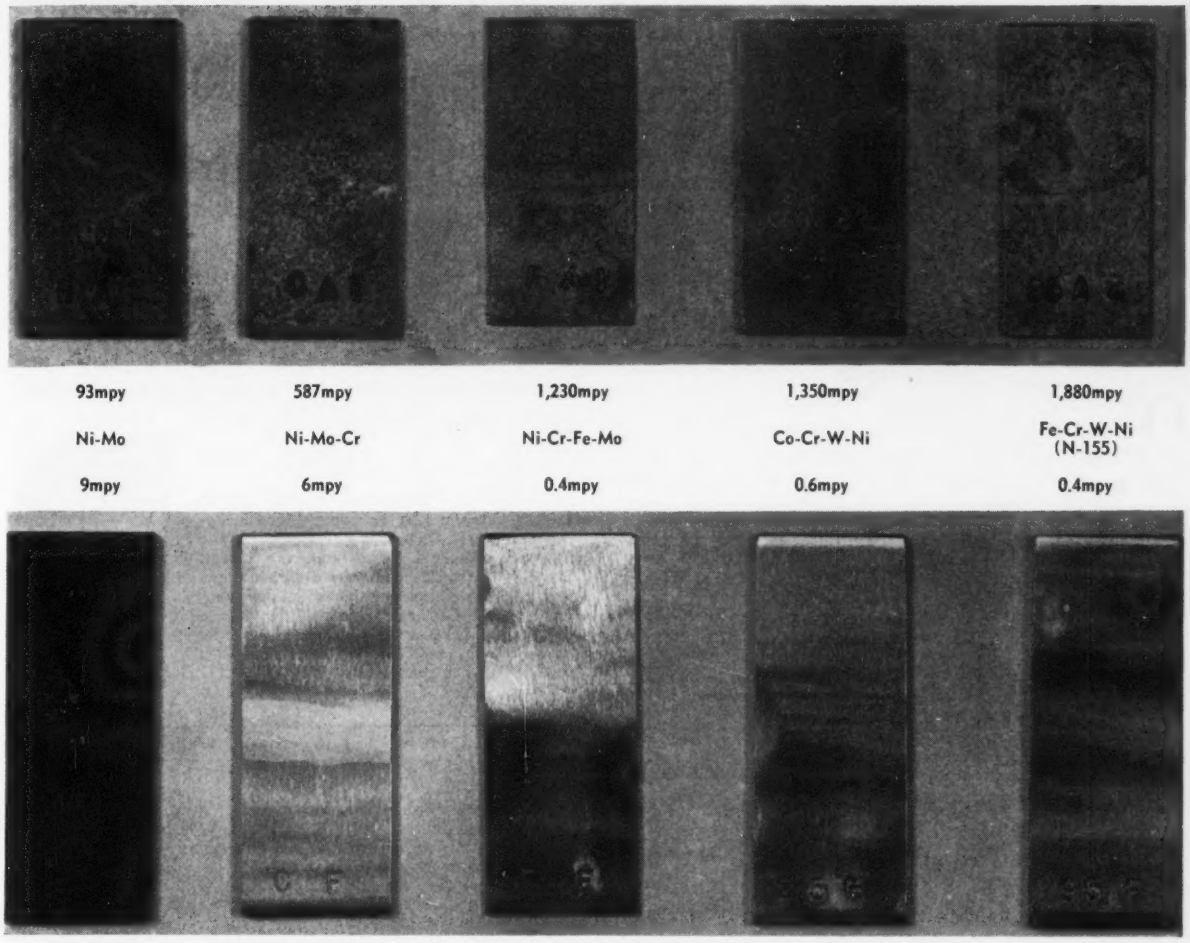

Figure 17—Increased corrosion of nickel-molybdenum alloy incurred above normal boiling point. Top row of samples were exposed to solution at 340 F, Ca psi—unwelded. Bottom samples were exposed to boiling solution (216 F)—as welded.

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Any discussions of this article not published above will appear in the December, 1957 issue.

Quantitative Comparison of Surface Finishes And Heat Treatment of Stainless Steel With Respect to Corrosion by Jet Fuels*

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Abstract

Samples were made from free machining AISI 440C stainless steel. They were identically hardened and tempered at 16 different tempering temperatures. The samples were then given five different surface finishes (i.e., grit blast, chro honed, ball burnished, ground and carborundum blast). After initial weighing the samples were subjected to a corrosion environment of JP-4 fuel and water. The samples were removed at varying times, cleaned, and again weighed. Weight losses were compared for different tempering temperatures and different surface finishes.

From the test results it was concluded that: (1) This steel should not be heated above 400 C (750 F) for greatest corrosion resistance, (2) Corrosion is greater when no JP-4 fuel is above the water, (3) The order of corrosion resistance was due largely to surface roughness of the different finishes. This order, in decreasing corrosion resistance, is as follows: (a) Ground surface, (b) Ball burnished, (c) Carborundum blast, (d) Chro honed, (e) Grit blast. 6.2.5

Introduction

ON SEVERAL occasions it was found that 440C stainless steel corroded under the environment of JP-4 fuel(1) and water. This investigation was initiated to determine the effect of environmental and material variables on corrosion. Preliminary work revealed, in a qualitative manner, that the surface finish influenced the rate of corrosion. Furthermore, it was noted[1] that the varying tempering temperatures also influenced the corrosion of a similar type stainless steel. These two factors were considered in this investigation.

Procedure

Specimen Preparation

Four hundred disk shaped samples were machined from the same bar of 440C stainless steel. Chemical analysis of this material is given in Table 1. Each sample was 1¼ inch in diameter and ⅛ inch thick with a hole ⅛ inch in diameter drilled in it for support during the heat treating operation and corrosion tests (see Figure 1). All samples were stamped for identification.

The samples were all hardened at the same time. This was done by spacing the samples about ½ inch apart on several lengths of nichrome wire; the ends of the wire were connected to the sides of the furnace charging basket. In this manner the samples did not touch each other, nor did they touch the charging basket, and hence were identically heated in an air atmosphere furnace at 1025 C (1875 F) for a total time of 30 minutes and cooled.

In the tempering operation the samples were laid flat in a basket and heated for three hours at different temperatures. There were sixteen separate tempering temperatures ranging from 150 C (300 F) to 900 C (1650 F) in 50 C increments; the temperature variation was ± 5 C. The effect of tempering temperatures on hardness also was determined. Rockwell C hardness readings were taken on the ground samples after corrosion testing.

After the heat treating operation the specimens were shot blasted to remove the scale and were then passivated to remove any trace of imbedded iron shot. All passivation that was done in this experiment was performed by immersing the samples in a solution for five minutes. This solution contained 50 percent (by volume) of 40 degree Bé nitric acid and 50 percent (by volume) tap water plus 2 percent (by weight) of sodium dichromate and was held at 62 C (145 F) ± 3 C. After the samples were removed they were rinsed in cold running water, dipped in hot water, and dried by blasting with a jet of air.

The five types of surface finishes were then applied to the samples:

1. Grit Blast—A No. 60 steel grit was used.
2. Chro Hone—A liquid honing or vapor blasting process (water + SiO_2 base abrasive).
3. Ball Burnishing—A tumbling operation per-

★ Submitted for publication September 6, 1956. A paper presented at the Thirteenth Annual Conference, National Association of Corrosion Engineers, St. Louis, Missouri, March 11-15, 1957.

(1) A kerosene-gasoline mixture (ANF-32).

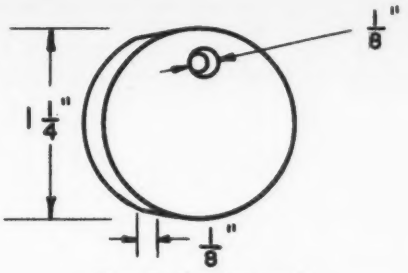

Figure 1—Corrosion sample.

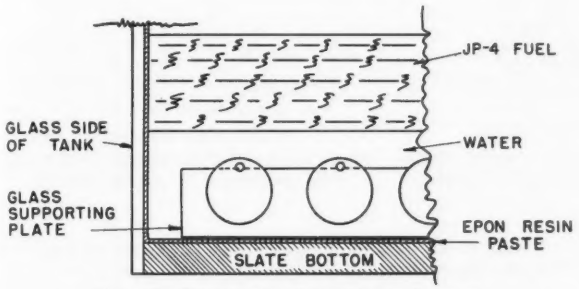

Figure 2—Cross section of corrosion environment.

TABLE 1—Chemical Analysis of Test Sample Material

ELEMENT	Weight Percent
Carbon	1.03
Chromium	17.03
Manganese	.35
Phosphorous	.020
Sulfur	.027
Silicon	.43
Molybdenum	.14
Selenium	.17

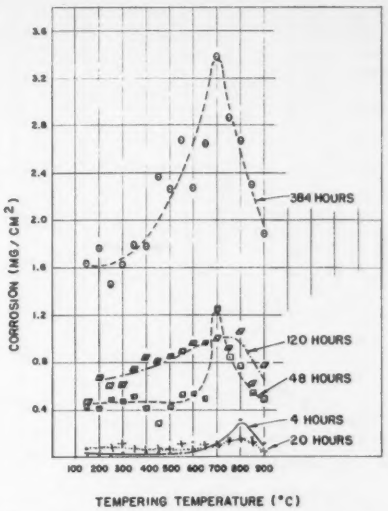

Figure 3—Corrosion weight loss versus tempering temperature for grit blast samples.

TABLE 2—Surface Roughness Values for Finishes

SURFACE FINISH	Surface RMS (Avge.)
"A" Grit Blast	62—75
"B" Chro Hone	32—44
"C" Ball Burnish	28—36
"D" Grind	15—24
"E" Carborundum Blast	38—52

TABLE 3—Analysis of Water for Corrosion Test

Hardness	150 parts per million
Alkalinity	116 parts per million
Chloride	4 parts per million
pH	7.7
Dissolved Solids	340 parts per million

formed by the Frederich Gumm Chemical Company.

4. Grind—Samples were ground with aluminum oxide wheel while water was flushed over the surface.
5. Carborundum Blast—A No. 60 carborundum shot was used.

After the samples were finished by these five methods, they were again passivated, carefully dried, and wrapped in absorbent paper (Scotch toweling). Surface roughness values were obtained on a profilometer for the different finishes. These are recorded in Table 2.

Method of Testing

All corrosion tests were performed in a glass walled tank with a slate rock bottom. The tank was 24 inches long, 18 inches high, and 12 inches wide. An Epon resin paste was used to coat the interior surfaces to prevent leakage. A special glass rack was designed to fit into the tank and support the glass rods that held the specimens.

Tap water with analysis shown in Table 3 was used in this experiment. The JP-4 fuel used in this testing was obtained from the same batch of fuel and stored in 5 gallon fireproof drums. It was found that the sulfate was less than 20 ppm, and the sulfite was less than 0.1 ppm. The acid content was found to be nil.

The procedure for the corrosion testing was as follows:

1. Four and one half gallons of the water were poured into the tank.
2. Weighed specimens, held by glass rods, were placed in position on the glass supporting rack in the water.
3. Four gallons of JP-4 fuel were added meticulously so as to prevent splashing. The composite arrangement is shown in Figure 2.
4. At end of test period, specimens were removed, pickled in a solution of 30 percent (volumetric) nitric acid + distilled water at 25 C (± 5 C) for 150 seconds, rinsed in tap water, and dried in a desiccator for three hours.
5. Specimens were weighed with accuracy to ± 0.0002 grams.

Results and Discussion of Results

Surface Finishes and Corrosion Resistance

Figures 3, 4, 5, 6, and 7 are curves of the results,

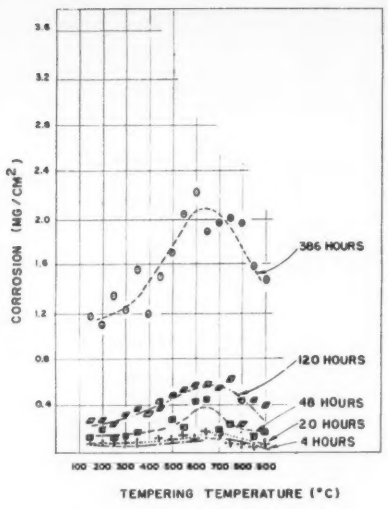

Figure 4—Corrosion weight loss versus tempering temperature for chro-honed samples.

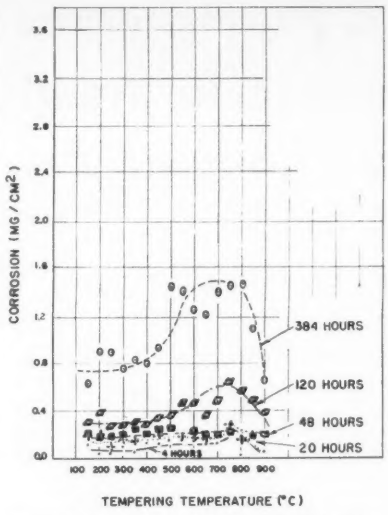

Figure 5—Corrosion weight loss versus tempering temperature for ball burnished samples.

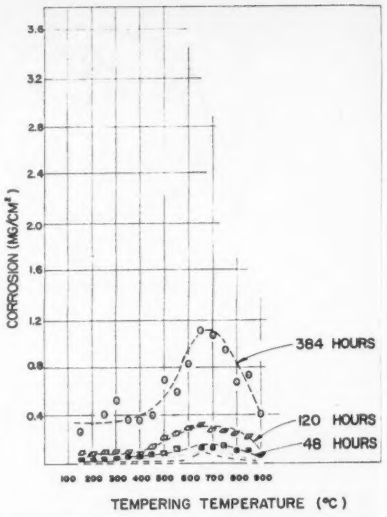

Figure 6—Corrosion weight loss versus tempering temperature for ground samples.

(i.e., weight loss (mg/cm²) versus tempering temperature.) The only variable that exists between these figures is the surface condition. Using the peak of the 384 hour curve (at about 700 C) as a criterion for corrosion, the surface finishes can be listed in order of their corrosion resistance with the ground samples representing unity. Note the order of corrosion resistance in Table 4.

The area available is a function of the surface roughness; this can be approximated as cones with varying heights with the area of the base of any single cone remaining constant. The area of a cone can be calculated from the equation:

$$\text{Area} = \pi r \sqrt{r^2 + h^2}$$

where r = radius of base of cone
$= 220 \times 10^{-6}$ in.
h = height of cone

The height of this cone can be obtained from the Peak to Valley values that are determined from Root Mean Square (RMS) values. These RMS values are measured with the use of a profilometer. A plot of the area available on one ordinate versus the RMS value is shown in Figure 9. Also in Figure 9 there is another curve of the maximum loss in weight (at 384 hours) versus the RMS values obtained for different surface finishes (Table 2). It can be noted that the Corrosion versus RMS curve is similar in shape to the Area Available versus RMS curve; from this it is apparent that the method of finishing the surface is not as critical as the surface roughness.

Hardness and Corrosion Resistance

Corrosion resistance is observed to decrease when the samples are tempered in the range of 400 C-850 C (750 F-1560 F). However, the corrosion peak occurs between 600 C-700 C (1110 F-1290 F). On comparing this latter temperature range with the hardness curve, Figure 10, it is noted that this corrosion peak occurs after the precipitation hardness, or secondary hardness maximum (450 C-500 C) has been exceeded. As a result of this, the loss of corrosion resistance of this metal possibly is attributed to the physical presence of a second phase that is not coherent with the matrix, similar to what has been hypothesized by Lula, Lena, and Kiefer,[2] and not to a coherent type of strain that has been suggested by J. J. Harwood[3] for other metals.

TABLE 4—Corrosion Resistance Factor (At Corrosion Peak)

FINISH	Loss in Weight Mg/cm²	Corrosion Resistance Factor
Grind	1.1	1.00
Ball Burnish	1.5	0.73
Carborundum Blast	1.9	0.58
Chro Hone	2.1	0.52
Grit Blast	3.4	0.32

Furthermore, the corrosion resistance increases above 800 C temper with a corresponding hardness increase. This is readily explainable since this steel is air hardening and the $\alpha \rightarrow \gamma$ transformation temperature (Ac_1) begins at 804 C.

Another point of interest can be observed in Figures 4, 5, 7, and 8. There appear to be two corrosion peaks. It is possible that this is experimental error. If, however, this actually is the case, two different, or more probably two simultaneous, mechanisms of corrosion occur.

Effect of the JP-4 Fuel on Corrosion Resistance

A comparison between Figure 7 (with JP-4 on surface) and Figure 8 (without JP-4 fuel) definitely indicates that the JP-4 fuel on the surface increases the corrosion resistance. One possible explanation for this is that the fuel will absorb the oxygen from the surrounding air and permit a generous supply to the water, which in turn tends to replenish the surface of the stainless steel with oxygen and continue a passive film. Another consideration is that oxygen

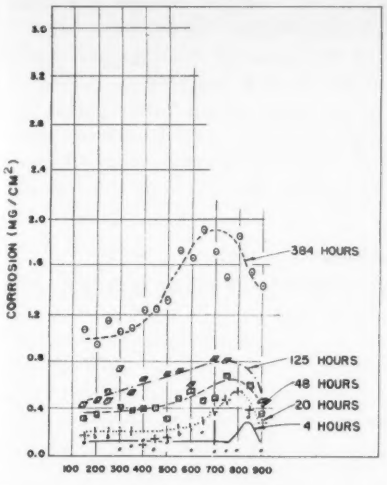

Figure 7—Corrosion weight loss versus tempering temperature for carborundum blast samples.

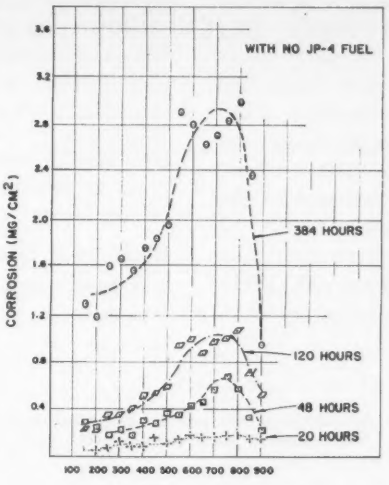

Figure 8—Corrosion weight loss versus tempering temperature for carborundum blast samples with No. JP-4 fuel above the water.

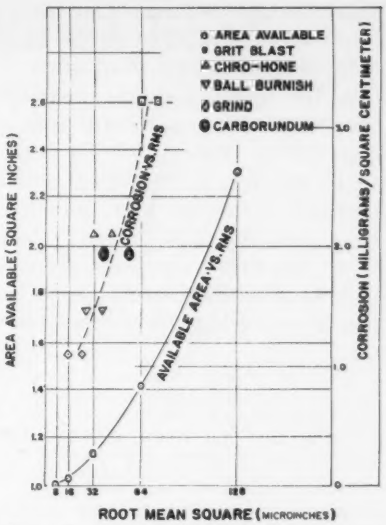

Figure 9—Area available and corrosion versus surface roughness (RMS—microinches).

is not present in the water in large enough quantities, because of higher solubility of oxygen in the fuel, to permit a continuous formation of corrosion products. Still another possibility is that the water dissolves something out of the fuel that inhibits corrosion.

Conclusions

1. Greater corrosion occurs when there is no JP-4 fuel above the water. This occurrence is not entirely explainable.
2. AISI 440 C and 440 F should not be heated above 400 C (750 F) if the best corrosion resistance is required. It can be noted that the greatest corrosion occurs when heated in the range of 600 C-700 C (1110 F-1290 F).
3. It is observed (Figure 9) that the two curves, Area Available versus RMS and Corrosion versus RMS, have the same curvature. From this it is concluded that the surface roughness is the primary cause for the variation in the amount of corrosion for the different surface finishes. Listed below, in order of decreasing corrosion resistance, are the different surface finishes:

    1. Ground Surface
    2. Ball Burnished
    3. Carborundum Blast
    4. Chro Honed
    5. Grit Blast

Acknowledgment

The author wishes to thank J. F. Quinlan and D. S. Stewart for their help, N. O. Bliss for his encouragement, and B. A. Schuller and R. L. Conn for their support of this corrosion project. Further acknowledgment must go to D. Caplan and Dr. A. A. Burr of Rensselaer Polytechnic Institute for the constructive criticism in preparing this material.

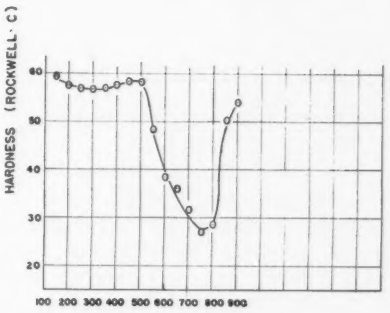

Figure 10—Hardness versus tempering temperature.

References

1. F. Kenneth Bloom. Effect of Heat Treatment and Related Action on Straight Chrome Stainless Steel. *Corrosion*, **9**, 56 (1953) Feb.
2. R. A. Lula, A. J. Lena and G. C. Kiefer. Intergranular Corrosion of Ferritic Stainless Steels. *Trans. American Society for Metals*, **46**, 1954.
3. J. J. Harwood. The Influence of Stress on Corrosion. *Corrosion*, **6**, 249-259 and 290-307, (1950) Aug. and Sept.

DISCUSSION

Questions by M. G. Fontana, Ohio State University, Columbus, Ohio:

1. Why were the specimens "passivated" before and after grinding?
2. If the specimens were "passivated" before exposure to the fuel environment, why did corrosion of any of the specimens occur?

Replies by R. G. Carlson:

Regarding Dr. Fontana's first question, after heat treatment the oxidized samples were shot blasted; to remove any imbedded iron particles (from the steel

shot) the samples were passivated prior to grinding. Passivation after grinding was done to simulate procedures used in the production of the parts made from this material. In answer to Question No. 2, this stainless, 440C, is not a true stainless because of the high carbon content. Similar high weight losses for a 410 stainless in 20 percent salt fog tests were reported in Reference 1 (F. Bloom). Furthermore, the steel contained 0.17 percent selenium. As noted by S. P. Watkins (Corrosion Resistance of Free Machining Stainless Steel. *Metal Progress*, **39**, 1941), this material has lower corrosion resistance.

Question by David Roller, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio

Was a mathematical treatment of the data performed to determine the effect of each variable on corrosion rate after removing the effect of the other variables? Were true statistical differences noted between samples given identical heat treatments and different grinding treatments taking into account the number of specimens?

Reply by R. G. Carlson:

A statistical analysis of the data was not performed.

Any discussions of this article not published above will appear in the December, 1957 issue.

DISCUSSIONS ON TECHNICAL ARTICLES ARE SOLICITED

Discussions on technical articles published in Corrosion will be accepted for review without invitation. Discussions must be constructive, accompanied by full substantiation of fact in the form of tables, graphs or other representative data and be submitted in three typewritten copies.

Authors of discussions are asked to supply one copy of figures suitable for reproduction and will be sent on request a copy of the NACE Outline for the Preparation and Presentation of Papers.

Discussions will be reviewed by the editor of Corrosion and will be sent to the author of the paper discussed for his replies, if any. Publication will be in the Technical Section with full credit to the authors together with replies. Discussions to papers presented at meetings of the association may be submitted in writing at the time of presentation or later by mail to the editorial offices of Corrosion, 1061 M & M Bldg., Houston 2, Texas.

The Growth of Ferrous Sulfide on Iron*

By R. A. MEUSSNER[1] and C. E. BIRCHENALL[2]

Abstract

The rate of growth of ferrous sulfide on iron in sulfur vapor was studied between 650 and 900 C with sulfur vapor pressures between 10 and 500 mm of mercury. When differences in conditions are taken into account, the rates seem to be substantially higher than those found by Hauffe and Rahmel. The behavior of inert markers and the sulfidation of eutectic mixtures of iron and ferrous sulfide indicate that the iron ion is much more mobile in ferrous sulfide than is the sulfide ion. Self-diffusion coefficients for the iron ions were calculated approximately, assuming that Wagner's theory of scale growth is applicable.

The markers were often found a considerable distance from their expected location at the metal-sulfide interface. This has been attributed to cracking at the interface in some cases and to interference with growth by the markers in other cases. It has been shown that nonreactive inclusions slow down the rate of sulfidation. 3.2.3

Introduction

THE FORMATION of a sulfide scale on iron is of great practical and theoretical interest because of the rapidity with which the sulfide grows at moderate to high temperatures. Consequently sulfide corrosion of steels in petroleum refining is an important problem, and the need for better understanding and control of the process is becoming more apparent. A number of rate studies have been made in recent years, covering a wide range of conditions. Dittrich[1] sulfidized iron, iron-nickel, iron-chromium, and iron-nickel-chromium alloys in the temperature range 300 to 500 C in hydrogen-hydrogen sulfide mixtures of varying ratio up to total pressures of 75 atmospheres. Comparative studies of a wide variety of plain carbon and alloy steels and surface treated steels in hydrogen-hydrogen sulfide atmospheres up to about 600 C were carried out recently by Backensto, Drew, and Stapleford.[2] In 1951 Dravnieks[3] measured the rates of reaction between iron and molten sulfur between 300 and 446 C.

The study of Hauffe and Rahmel[4] in 1952 of the reaction of iron with sulfur vapor included sulfur pressure as a variable but was limited to a narrow temperature range. The present experimental conditions are most like the last, although several modifications in procedure have been made. Some of the changes relate to possible sources of error in Hauffe and Rahmel's measurements, although their descriptions are not sufficiently detailed to estimate the magnitude of uncertainty to be expected.

Equilibrium studies of the binary system iron-sulfur indicate that two stable sulfides are to be expected at high temperatures. Pyrite, FeS_2, has a fairly high dissociation pressure, and the reaction conditions can be adjusted easily to avoid its formation. Pyrrhotite, also called ferrous sulfide or FeS, has a wide range of stable composition, from nearly stoichiometric on the iron rich side to almost 20 percent deficiency of iron ions on the sulfur rich side at high temperatures. The phase has the NiAs structure and appears to be stable down to room temperature with only some magnetic and order-disorder transitions.

Ordering of the cation vacancies was demonstrated by Bertaut[5] using X-ray diffraction. This sulfide is very brittle, but not very strong, an unhappy combination which undoubtedly intensifies the corrosion problems associated with this material, in addition to increasing the difficulty of studying it experimentally.

Experimental Procedure

The rate of reaction of iron and sulfur vapor to form pyrrhotite was determined by measuring the quantity of iron converted to sulfide as a function of the reaction time. The equipment used in these reaction rate studies is shown in Figure 1. The bulb of the Vycor reaction vessel which contained the iron specimen was maintained at the desired reaction temperature by furnace F, and the desired sulfur vapor pressure over the specimen was attained by immersing the sulfur bulb of the cell in a small bath of molten salts (P).

An electrically heated bridge covering the tube connecting the iron bulb to the sulfur bulb produced a temperature distribution in the reaction cell which continuously increased from the constant temperature region of the sulfur bulb to that of the iron bulb. The temperatures of the sulfur and iron bulbs which were automatically regulated by potentiometric controllers were monitored during each experiment through thermocouples T_S and T_F. These measuring thermocouples were calibrated in place by comparison with calibrated standard couples placed in a dummy reaction vessel. The temperatures in the bulbs were uniform to within $\pm\frac{1}{2}$ degrees C and constant in time to within ± 1 degree C. The temperature required to produce the desired sulfur vapor pressure in the cell was calculated from the data of K. K. Kelley.[6] On the basis of these data the temperature variation of the salt pot (± 1 degree C) produced variations in the sulfur vapor pressure amounting to ± 4 percent at 10 mm and ± 2 percent at 100 mm.

★ Submitted for publication October 29, 1956.

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(2) Associate professor of chemistry, James Forrestal Research Center, Princeton University, Princeton, New Jersey.

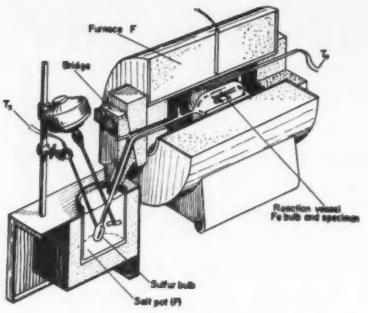

Figure 1—Experimental apparatus.

The iron specimens, small rectangular plates, were drilled to accommodate the two ceramic support rods and surface ground to assure a uniform thickness. These specimens were approximately 7 mm wide by 19 mm long, and ranged from 0.6 mm to 2.5 mm in thickness.

In order to relieve the machining stresses, decarburize the material, and reduce surface oxides, the iron specimens were subjected to successive prolonged treatment at 750 to 900 C in atmospheres of purified wet and dry hydrogen. Three types of iron, designated as A, P, and C, were used in these studies. Iron A, Armco iron, contained a moderate population of small elongated nonmetallic inclusions. While these were not reduced during the hydrogen firing, they were spheroidized. Iron P was found to contain a large number of nonmetallic inclusions. These appeared as a very high population of small spheroidal particles and a number of rather massive stringers. Because of this, iron P was used in only a few experiments. Iron C, a relatively pure iron which was almost entirely free of inclusions, was used for the major portion of this work. The chemical analysis of this material indicates impurities in the following concentrations: Ni and Mn each less than 0.01 percent, Si and P each less than 0.005 percent, and Mg, Cu, Al, Sn, and S each less than 0.001 percent. Carbon was undetectable.

The sulfur was chemically pure flowers purified in the laboratory by refluxing over magnesium oxide —the technique of Bacon and Fanelli.[7]

Preparation of Reaction Cell

The preparation of each reaction cell was a rather involved process. After the vessel had been made from Vycor tubing, an iron specimen was selected, its thickness accurately measured with a micrometer caliper, clean ceramic support rods inserted in the holes of the specimen, and this assembly slipped into the open iron bulb of the Vycor cell. On some specimens thin lines of chromium oxide ("CRO" polishing abrasive) were drawn across the width of the 7 by 19 mm faces to serve as inert markers. In order to prevent the oxidation of the iron during the sealing of the Vycor bulb, large pieces of resublimed dry ice were placed around and in front of the specimen. This provided an excellent radiation shield, for the iron surfaces appeared bright and clean after the cell had been sealed into a vacuum system and the remaining dry ice was pumped off. The entire cell was gently flamed, the sulfur slowly melted, and then heated to boiling under the reduced pressure of the system. By careful manipulation of the torch during these flaming operations, and during the subsequent cooling period, it was possible to control the condensation of the sulfur vapor in the cell and to keep the iron bulb free of sulfur. With the sulfur thoroughly degassed, the iron sample clean and free of sulfide, and the pressure in the cell below 5×10^{-5} mm of mercury, the cell was sealed off.

The procedure of the actual experimental run was simple. When the furnace and salt pot were stabilized at the desired temperatures the bridge was momentarily removed and the iron bulb of a reaction vessel was inserted into the furnace. The sulfur bulb was refrigerated in a dry ice pack until the furnace regained the desired temperature and then the salt pot was brought into position around the sulfur bulb to start the run. Throughout the reaction period the temperatures of the iron and sulfur bulbs were carefully monitored and minor control adjustments made to maintain these temperatures at the desired values. After the specified reaction time the salt pot was lowered, the sulfur bulb quickly chilled in an air blast, the bridge removed, and the reaction cell withdrawn from the furnace. The cell was then permitted to air cool to room temperature.

Temperature Increase

During the first few experiments it was observed that the temperature of the iron bulb, as measured by thermocouple T_F, increased a few degrees shortly after the salt pot was raised into position. This was due to the heat generated by the initial reaction of the clean iron surfaces with the sulfur vapor. The magnitude of the true temperature rise experienced by the specimen in this initial period of the reaction was first indicated directly by the results of an exploratory experiment designed to measure the rate of the reaction with the iron in the austenitic state. In this experiment the iron temperature was 970 C; sulfur pressure was 100 mm of mercury. The reaction product was molten iron sulfide indicating that a temperature of 988 C (the Fe:FeS eutectic temperature) had been exceeded.

In order to gain a more meaningful measure of the temperature of the iron during this initial reaction period, several cells were prepared in which the iron specimens were not supported on ceramic rods but lay instead on the bottom of the iron bulb. A fine wire thermocouple was attached to the Vycor vessel immediately beneath the center of the specimen and the temperature at this point measured during the reaction period. In this series of experiments the sufur vapor pressure was held constant at 100 mm of mercury. With a specimen temperature of 650 C the maximum rise was approximaely 10 C. At 800 C the rise was about 25 degrees C, and in some cases at 900 C the maximum temperature exceeded the eutectic temperature—a temperature rise of 88 degrees C. Since this heat effect was due to a reaction at the surface of the iron, it was to be expected

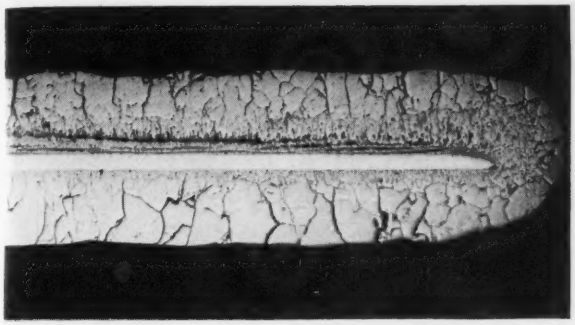

Figure 2—Iron C reacted at 800 C and 100 mm P_{S_2} for three hours showing longitudinal cracks in the FeS along one side of the specimen. (½ of entire specimen.) 7.5X, bright field.

that the magnitude of the temperature rise would be greatly affected by the surface to volume ratio of the specimen, as shown by samples reacted at 900 C and a sulfur pressure of 100 mm of mercury. The reaction product of one of two iron specimens of 1.77 grams mass was molten while samples of mass greater than 2 grams showed no evidence of a liquid phase being formed during the reaction. Thus, it would appear that the specimens selected for long reaction times (i.e., thick specimens), should yield the more reliable data since the initial temperature rise would be minimized. However, the formation of cracks and the separation of the scale from the metal substrate in these heavier specimens also introduced additional complications. This aspect of the problem will be discussed more fully in a later section on the microstructural examination of these specimens.

In all cases studied the period of overheating was of short duration. The specimen temperature rose to its maximum value within one minute after the salt pot was elevated and dropped quickly thereafter. After five minutes the overheat of the specimen had been reduced to one-half of its maximum value and within ten minutes the temperature was within two degrees of the desired reaction temperature. Throughout the remainder of the reaction period the temperature variation of the iron was seldom greater than ±1 degree C.

Since the rapid rise in the specimen temperature immediately following the immersion of the sulfur bulb in the salt pot indicated the initiation of the reaction, the reaction time has been taken as the total elapsed time between raising the salt pot and the removal of the reaction cell from the furnace. As a consequence of these observations of the variations in the specimen temperature during the initial period of the reaction, the data from specimens reacted for less than one hour were not considered in the evaluation of the reaction rates. That substantially no sulfide was formed on the iron until the sulfur was heated was determined by holding a reaction cell at 800 C for 45 minutes without heating the sulfur bulb, extracting the cell from the furnace, and examining the iron specimen. The iron surfaces were only slightly darkened by the formation of a very thin sulfide film.

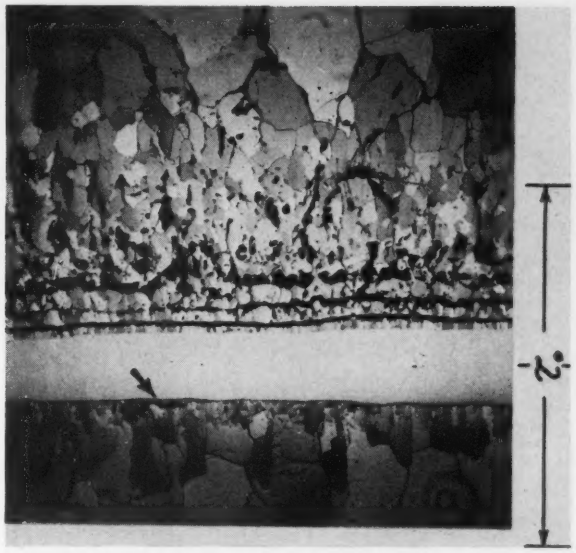

Figure 3—Same specimen as Figure 2, showing longitudinal cracks and the location of markers (arrows) on each side of the iron. τ_o indicates the original thickness of the iron specimen, 35X, polarized light.

Microstructures

A series of photomicrographs illustrating the structures observed in these specimens are shown in Figures 2 through 10. Because of the friable nature of the sulfide scale it was essential that the specimens be imbedded in a suitable mounting material before sectioning for metallographic study. A liquid epoxy resin (Epon 828 with triethylenediamine as a catalyst) was used. Satisfactory mounts were obtained by vacuum impregnating the specimens with this plastic—after carefully breaking away the corners of the sulfide layer to admit the plastic to the internal cracks, and then baking at 60 C for several hours.

With the exception of Figures 7 and 8 (i.e., the Fe:FeS eutectic specimens), all of these photomicrographs illustrate the general features of the sulfidized specimens. One or more longitudinal cracks frequently formed in the sulfide along one side of the iron plate. The surface of the iron sulfide layer was made up of very large grained material while the region near the Fe/FeS interface was rather fine grained. In the course grained region many intergranular cracks developed. (In polishing, some particles of sulfide were pulled out of this very brittle material. These voids are readily differentiated microscopically from true pores).

The fine grained region, extensive on the side of the specimen showing longitudinal cracking and rather limited on the opposite side of the iron, contained a large number of fine pores. The oxide markers (indicated by arrows in those prints where their presence is not immediately obvious) are found immediately adjacent to the Fe/FeS interface (Figure 6), only slightly removed from this interface (Figures 3, 4, and 5), or deeply imbedded in the sulfide layer (Figures 3 and 5). Markers at or near the Fe/FeS interface were observed on the "sound" side

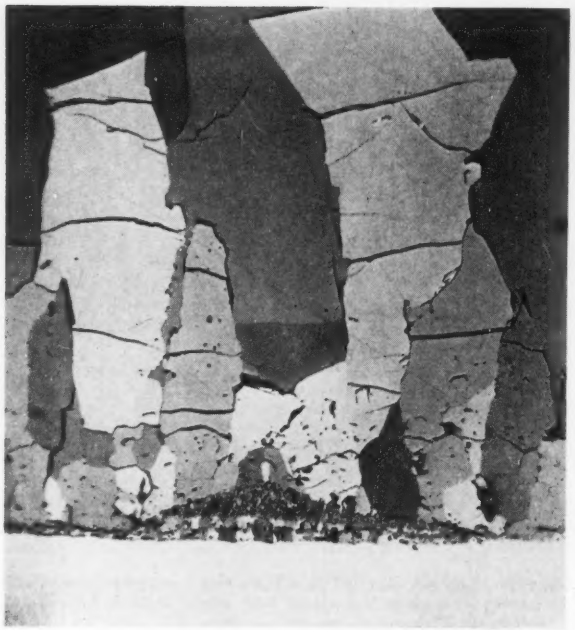

Figure 4—Same specimen as Figure 2, showing one of the markers located very near the Fe/FeS interface in the "sound" side of the specimen. 200X, polarized light.

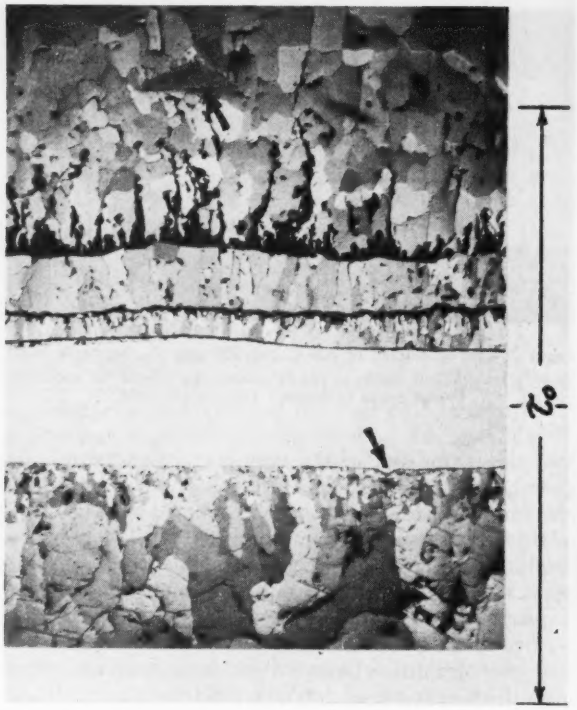

Figure 5—Iron C reacted at 800 C and 100 mm P_{S_2} for 5½ hours, showing deep fissuring along the longitudinal cracks and the relative location of markers (arrows) on the two sides of the specimen. τ_o indicates original thickness of the iron. 40X, polarized light.

of the specimen while those far removed from the interface were seen only where a series of longitudinal cracks were present—though the markers appear well beyond the last visible crack and are located in an apparently sound sulfide matrix.

The width of the longitudinal cracks increased during the cooling of the specimen because of the relatively larger coefficient of thermal expansion of the iron. To minimize this effect and to avoid severe cracking of the sulfide arising from this dimensional change, the thickness of the original iron was adjusted so that the residual plate in the specimen would be thin—approximately 0.5 mm.

Iron Ion Is Mobile Species

Before discussing the formation of these structures in detail, it is desirable to establish the fact that the iron ion is the mobile species in the growth of the iron sulfide layer (i.e., that iron migrates through the sulfide from the Fe/FeS interface to the $FeS/S_2(g)$ interface) and that the growth of the layer is due to the formation of FeS at this latter interface. The observation that some of the inert markers do remain at the Fe/FeS interface is sufficient to prove this point.

Many markers were observed at or very close to this interface. This is illustrated in Figure 6 where a marker is seen to have remained at the Fe/FeS interface while 70 percent of the original thickness of the iron specimen has been converted to FeS by growth at the $FeS/S_2(g)$ interface. However, additional proof of this point is illustrated in Figures 7 and 8. These photomicrographs show the effect of exposing an Fe:FeS eutectic structure to a sulfidizing atmosphere. This specimen resulted from the attempts to study the reaction at temperatures above 900 C, where the initial reaction proceeded at such a high rate that the specimen temperature was raised well above the eutectic temperature. The resulting liquid flowed into the bottom of the Vycor bulb, and froze, and during the remainder of the reaction time this eutectic material was exposed to the sulfur atmosphere (100 mm of mercury). As shown in Figures 7 and 8 the iron of the eutectic iron particles near the exterior interface has migrated through the sulfide to the $FeS/S_2(g)$ interface leaving behind voids of the same size and shape as the remaining iron particles more distant from the interface. Thus it must be concluded that the sulfide ion is much less mobile than the iron ion and that the latter is largely responsible for the growth of the sulfide.

The increase in grain size of the sulfide near the outer surface of the normal specimens may be interpreted as a result of this growth mechanism. The initial layer of FeS nucleated on the metal is fine grained but because of the anistropy of the growth rates of the sulfide crystal surfaces exposed to the atmosphere some grains are quickly enveloped by their fast growing neighbors and a coarse grained structure results.

A careful study of the microstructures of a large number of specimens has suggested the following sequence for the formation of the longitudinal cracks. Early in the sulfidation period the iron specimen is confined in a compact, fairly rigid shell of iron sulfide. As the thickness of this shell increases the vol-

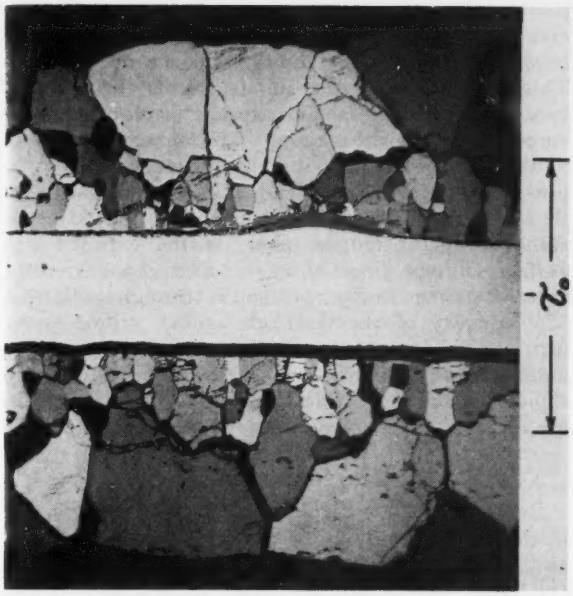

Figure 6—Iron C reacted at 800 C and 100 mm P_{S_2} for 15 minutes showing a marker at the Fe/FeS interface. τ_o indicates the original thickness of the iron. 100X, polarized light.

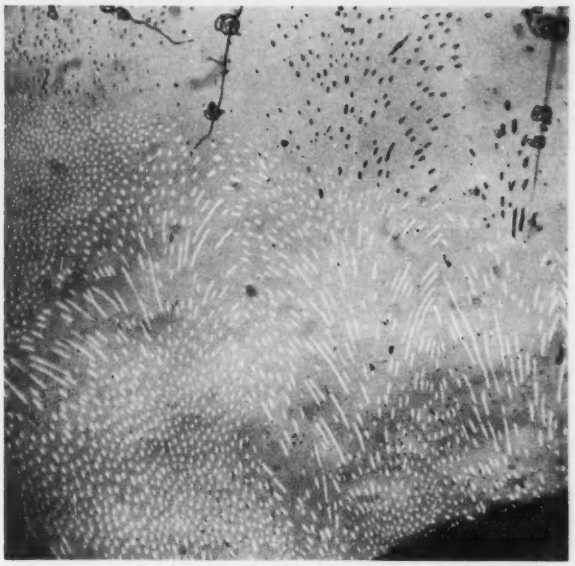

Figure 7—Eutectic Fe:FeS specimen. Iron C sulfidized at a temperature above 900 C at 100 mm P_{S_2}. The sulfur vapor interface is above; the iron substrate below the area of this photomicrograph. Voids resulting from the migration of iron to the FeS/S_2(g) interface are shown in the upper portion of the picture. 200X, bright field.

ume of the remaining metal must decrease. Because of the geometry of the specimen and the growth mechanism, compressive stresses arise in the outer layers of the sulfide and tensile stresses across the Fe/FeS interfaces. Under this stress system the sulfide appears to resist deformation. As the sulfide increases in thickness the magnitude of these stresses increases until failure occurs at the Fe/FeS interface. Since the rate of removal of iron is greatest at the corners and edges of the specimen, the crack generally originates near these sites and spreads slowly across the iron interface. The formation of such a crack on one side of the iron seems to be sufficient since such cracks are generally observed in the sulfide on only one side of the specimen. A new layer of sulfide grows on the exposed iron and in time the process of the formation of a longitudinal crack is repeated as shown in the photomicrographs.

Measurements of the thickness of the sulfide layers on opposite sides of the iron specimen indicate that this cracking apparently does not materially alter the growth rate. The thickness of the cracked sulfide layer was never more than 10 percent greater than that of the relatively sound layer. This small difference is not considered important in view of the difficulty of the measurement. The source of these difficulties (i.e., the unevenness of the outer surface, and the presence of void volumes within the scale layer) can be seen in Figure 2. From these observations it seems that the overall rate of growth of the sulfide layers on opposite sides of the iron specimen was unaffected by the complications of crack formation. Hence, it is reasonable to consider the rate of removal of iron to be the same from both faces of the specimen. On this basis the center line of the remaining iron plate is considered to coincide with that of the original specimen and the thickness of the original plate (τ_o) has been so indicated to the right of Figures 3, 5, and 6. The location of the markers on the cracked side of these specimens indicates that these markers were detached from the Fe/FeS interface early in the reaction period.

Detachment of Markers

A more detailed examination of the microstructures suggests two possible means for the detachment of markers under the conditions that the iron ion alone is mobile. The markers may become detached by the action of a crack propagating along the Fe/FeS interface as previously suggested. In Figure 4 it appears that a crack has penetrated between the marker and the iron substrate late in the reaction period.

Another mechanism for the detachment is shown in Figure 5. Here the small marker shown in the lower right of the picture has become detached as a result of its interference with the flow of iron from the area of attachment. By impeding the reaction the marker sits on a protruding bit of iron as seen in Figure 6. This base is undercut as the reaction proceeds and finally the marker is completely detached from the iron surface without crack formation. The two "deeply imbedded" markers of Figures 3 and 5 may have become detached by the undercutting mechanism. In the latter cases the sulfide matrix about these markers is sound and evidence of an old crack is lacking. Also the iron surface beneath these markers is slightly convex, perhaps indicating the last remains of the "stump" from which the markers were finally detached.

It is believed that as a consequence of the com-

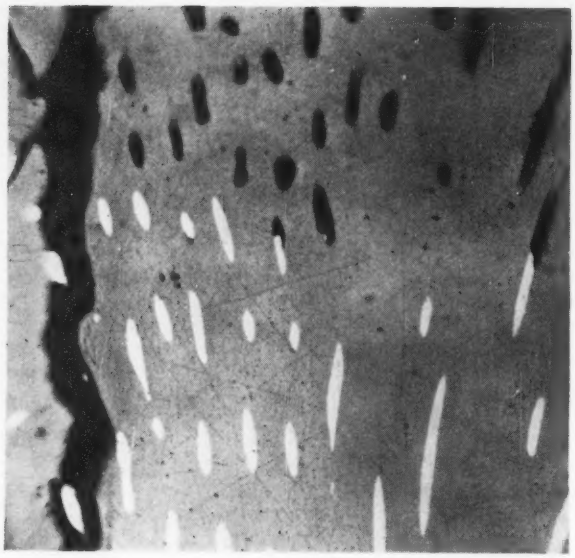

Figure 8—Same as Figure 7 showing voids resulting from the migration of iron from the eutectic iron particles to the $FeS/S_2(g)$ interface. Transverse crack at left is along a grain boundary. 750X, bright field.

pression stresses near the surface, the longitudinal cracks are not connected to the exterior surface of the sulfide layer and hence the direct entry of sulfur vapor into these voids is not possible. The observations supporting this view are (1) the equality of the total amount of sulfide formed on the cracked and uncracked sides of the specimen, and (2) the unusual pore or fissure formation on the inside of the detached sulfide layer.

The transverse cracks, most often intergranular cracks, are presumed to form on cooling and not during the reaction; therefore these cracks cannot operate to admit sulfur vapor to the interior of the specimen. The transverse crack shown in Figure 8, the eutectic specimen, clearly illustrates that no penetration of sulfur vapor has occurred along this grain boundary crack since iron particles appear very near, indeed appear to be in the crack as a result of the broadening of the crack in polishing. Thus it appears that this type of crack forms on cooling.

One possible explanation for the formation of the fissures and the growth of the new layer is the following. The formation of a longitudinal crack separates the FeS layer from the source of iron. Iron ions continue to migrate toward the $FeS/S_2(g)$ interface, (i.e., down the existing concentration gradient). As a result iron ions continue to arrive at the external surface and react to form additional sulfide. The sulfur concentration, and the iron vacancy concentration, at any point in the interior of the sulfide layer is thus increased.

Sulfur Content

Hägg and Sucksdorff[8] have shown that an increase in sulfur content produces a marked contraction of the lattice. Since the sulfur concentration at the exterior surface remains constant no contraction is experienced there, but the extent of the contraction due to the possible change in composition increases with distance beneath the gas interface—becoming large at the interior surface of the layer. This contraction of the sulfide causes the crack to broaden and may also initiate the intergranular fissures. As the sulfur content of the material adjacent to the crack increases, the equilibrium sulfur vapor pressure also increases, and sulfur is released into the crack volume. By this means a new layer of sulfide is permitted to grow on the exposed iron surface without direct entry of sulfur vapor from the exterior or the transport of sulfur through the lattice. The equality of the thickness of the sulfide layers on the two sides of the specimen might be taken to indicate that sulfur is vaporized and transferred so rapidly across the gap that the rate controlling process remains diffusion of iron ions in the solid sulfide.

Rate Measurements

Because of the difficulties in determining the true thickness of the sulfide layers (i.e., rough surface, and interior pores and cracks) measurements of scale thickness were abandoned and the progress of the sulfidation reaction was determined by measuring the decrease in the thickness of the iron specimen. These measurements were made near the center of the plane of polish. By this means corner and edge effects, which must be present in any measurement such as total weight change, were entirely eliminated. The measurements were made on a projection microscope and the recorded values of the iron thickness were the average of ten determinations spaced uniformly across a region approximately 5 to 7 mm long near the center of the specimen. One-half of the decrease in thickness of the iron plate ($\frac{1}{2}\ \Delta\tau$) is then a measure of the mass of iron per unit area of interface that has been converted to sulfide during the reaction period.

The data from specimens of pure iron, iron C, isothermally reacted at a sulfur vapor pressure of 100 mm of mercury with temperatures from 650 to 900 C are shown graphically in Figure 11. Complete series of specimens were employed to define the course of the reaction at 700 and 800 C, while only a few specimens were used to indicate the reaction rates at 650, 750, and 850 C. Because of the magitude of the overheating during the initial period of the reaction only two specimens survived the experiments at 900 C without showing evidence of the formation of a liquid phase—and in one of these the sulfur reservoir was almost entirely empty at the end of the experiment. Thus the line for the 900 C reaction rate is shown as a dashed line. Investigations were not pursued below 650 C because a second sulfide, pyrite (FeS_2), becomes stable under these conditions.[9] This plot of iron consumed per square centimeter of interfacial area vs the square root of the reaction time clearly indicates that the parabolic growth law is obeyed. Thus the sulfide layer is "protective" and the growth rate is determined by the diffusion of iron through the scale. The fact that these data extrapolate rather well to zero iron consumption at zero reaction time confirms the observa-

tion that the temperature rise during the initial period of the reaction was short in duration.

Pressure Dependence

In addition to the experiments defining the temperature effect on the reaction rate a second series of experiments was conducted to study the pressure dependence of the rate. In these experiments the iron temperature was maintained constant at 800 C, a reaction time of two hours was employed, and the sulfur vapor pressures were varied from 500 down to 10 mm of mercury. The data from these experiments are given in Table 2 and are shown graphically in Figure 12, as Wagner's rational rate constants.[10] Data from the first set of specimens prepared in exactly the same manner as the specimens used in the temperature dependence experiments are plotted as filled circles. These data, in the pressure range of 500 to 50 mm of mercury indicate the theoretically predicted pressure dependence of the reaction rate. At a sulfur vapor pressure of 25 mm of mercury the observed reaction rate was lower than expected, and at 10 mm of mercury the rate was negligible (the specimen survived the two hour exposure without forming much more than a tarnish film).

A subsequent attempt to investigate the rate at this pressure yielded a specimen with a scale of nonuniform thickness. The sulfide layer was thickest on the end of the iron specimen near the sulfur inlet and tapered off to a tarnish film at the opposite end. The scale at the thick end was never measured but was estimated to be approximately ⅓ mm in thickness. These results suggested that some gas other than S_2 was present in the cell during the reaction period and that the gas was of lower molecular weight than the sulfur vapor and thus segregated into the hot iron bulb by thermal diffusion. This gas effectively lowered the sulfur vapor pressure of the atmosphere surrounding the iron specimen and markedly reduced the rate of the reaction. After reaction, a Tesla coil discharge indicated a pressure of the order of 10^{-3} to 10^{-4} mm of mercury pressure in the cells. When the sulfur bulb of an unopened cell was immersed in liquid nitrogen the gas discharge slowly weakened and finally vanished. On opening the cells a very faint odor was occasionally detected. These observations indicated that the gas in the cell was probably H_2S—the hydrogen source being the iron specimen itself.

Minimizing of Thermal Diffusion Effect

In order to minimize this thermal diffusion effect two modifications in the preparation of the reaction cells were made: (1) the volume of the reaction vessel was increased by lengthening the iron bulb and by enlarging the diameter of the tube connecting it to the sulfur bulb, and (2) the hydrogen fired iron specimens were vacuum annealed at 800 C for eight hours at a pressure of approximately 10^{-5} mm of mercury. Although these modifications did not completely eliminate the problem, they did result in increased rates at low pressures.

The specimens subjected to the vacuum extraction process are indicated in Table 2 as "CV" specimens and are plotted as open circles in Figure 12. Specimens CV-1 and CV-5, reacted at 10 mm pressure,

TABLE 1—Calculation of Rational Rate Constants and Iron Self-diffusion Coefficients

P''_{S2} = 100 mm, (S') = 36.5 w/o, = ρ_{Fe} = 7.86 gm/cm³

Temp., Degrees C	K' cm Fe / cm² sec$^{1/2}$	S'' (w/o)	$\bar{S}$ (w/o)	ρ_{FeS} (gm/cm³)	K_r eq. Fe / cm² sec	—log P'_{S2}	Z''_{Fe}	$\bar{C}_v$ moles / cm³	$\bar{D}_{Fe}$* cm² / sec
650	3.33x10^{-4}	43.15	39.82	4.53	5.20x10^{-8}	8.72	2.65	5.63x10^{-2}	1.7x10^{-8}
700	4.33x10^{-4}	42.00	39.25	4.57	8.43x10^{-8}	7.77	2.53	5.62x10^{-2}	3.2x10^{-8}
750	5.42x10^{-4}	41.23	38.86	4.62	12.72x10^{-8}	6.94	2.44	5.60x10^{-2}	5.2x10^{-8}
800	6.50x10^{-4}	40.50	38.50	4.64	17.88x10^{-8}	6.19	2.38	5.58x10^{-2}	8.1x10^{-8}
850	8.00x10^{-4}	39.87	38.18	4.66	26.48x10^{-8}	5.52	2.31	5.56x10^{-2}	13.2x10^{-8}
900	9.33x10^{-4}	39.35	37.92	4.68	35.32x10^{-8}	4.87	2.27	5.55x10^{-2}	19.4x10^{-8}

Data of Hauffe and Rahmel[1]

P''_{S2} = 10 mm

Temp., Degrees C	$\left(\frac{K' \text{ gm S}}{\text{cm}^2 \text{ sec}^{1/2}}\right)^2$	S'' (w/o)	$\bar{S}$ (w/o)	ρ_{FeS} (gm/cm³)	K_r eq. Fe / cm² sec
620	0.65x10^{-6}	41.26	38.88	4.60	1.14x10^{-8}
644	0.82x10^{-6}	40.86	38.68	4.62	1.43x10^{-8}
670	1.11x10^{-6}	40.40	38.45	4.64	1.94x10^{-8}
697	1.60x10^{-6}	39.94	38.22	4.65	2.81x10^{-8}

TABLE 2—Pressure Dependence of Rate Constants

T = 800C, S' = 36.5 w/o, ρ_{Fe} = 7.86 gm/cm³

Spec. No.	P''_{S2} (mm Hg)	K' cm Fe / cm² sec$^{1/2}$	S'' (w/o)	$\bar{S}$ (w/o)	$\overline{\rho_{FeS}}$ (gm/cm³)	K_r eq Fe / cm² sec
C-7	500	6.85x10^{-4}	42.70	39.60	4.54	23.3x10^{-8}
CV-11	500	7.08x10^{-4}				24.8x10^{-8}
CV-13†	250	7.00x10^{-4}	42.00	39.25	4.57	24.8x10^{-8}
CV-4	100	5.37x10^{-4}	40.50	38.50	4.64	23.0x10^{-8}† 12.8x10^{-8} 17.8x10^{-8}**
C-3	50	6.23x10^{-4}	39.86	38.18	4.66	16.7x10^{-8}
CV-2	25	5.83x10^{-4}	39.30	37.90	4.69	14.2x10^{-8}
C-8	25	4.72x10^{-4}				9.33x10^{-8}
CV-1	10	3.47x10^{-4}	38.64	37.57	4.72	4.74x10^{-8}
CV-5	10	2.88x10^{-4}				3.27x10^{-8}

Data of Hauffe and Rahmel[1]

T=670 C

Spec. No.	P''_{S2} (mm Hg)	$\left(\frac{K' \text{ gm S}}{\text{cm}^2 \text{ sec}^{1/2}}\right)^2$	S'' (w/o)	$\bar{S}$ (w/o)	$\overline{\rho_{FeS}}$ (gm/cm³)	K_r eq Fe / cm² sec
	100	1.66x10^{-6}	42.70	39.55	4.55	2.88x10^{-8}
	10	1.11x10^{-6}	40.50	38.45	4.64	1.94x10^{-8}
	1	0.87x10^{-6}	38.80	37.60	4.72	1.53x10^{-8}
	0.1	0.59x10^{-6}	38.30	37.35	4.74	1.04x10^{-8}

* Data from Table 1
† Specimen temperature of CV-13 = 805 C starred data corrected to 800 C

Figure 9—Fe/FeS interface of an Iron A specimen (annealed in argon) reacted at 700 C and 100 mm P_{S_2} for seven hours, showing the accumulation of non-metallic inclusions in the FeS near the interface. 300X, polarized light.

developed rather thick uniform scales; however, the rates were less than expected from an extrapolation of the high pressure data. This may indicate that the volume of residual gas was still sufficiently large to interfere. At a pressure of 25 mm the rate was increased to the extrapolated value by these modifications. Vacuum extracted specimens were reacted also at sulfur vapor pressures of 100, 250 and 500 mm. The 500 and 250 mm samples yielded rates comparable with the earlier data. The 100 mm specimen yielded a rate lower than that established by the temperature dependence series of specimens reacted at 800 C. Because the rate at 800 C and 100 mm has been repeatedly established by the earlier experiments, the result from this vacuum extracted specimen is assumed to be in error.

Inclusions Reduce Rate

These data then define the reaction rate at 800 C over the range of sulfur vapor pressures from 500 mm to 25 mm of mercury. The decrease in rate at 10 mm is interpreted as being the effect of residual hydrogen reducing the effective sulfur potential of the atmosphere over the iron specimen.

The results of a few experiments run on irons A and P are presented in Figure 13. Iron A showed a slightly lower reaction rate than the pure iron C as seen by a comparison of the data from experiments at 700 C and a sulfur pressure of 100 mm of mercury. However, it is also to be noted that these data for iron A indicate a parabolic growth of the sulfide. This lower rate of reaction of A, as compared to C, may be readily associated with the occurrence of nonmetallic inclusions. Inclusions were deposited at the Fe/FeS interface by the consumption of iron during the reaction, and since they were insoluble in the sulfide, their presence at this interface effectively decreased the available interfacial area. Thus, the measured reaction rate was lowered.

A photomicrograph of the interface of one of these specimens is shown in Figure 9. In this the rather porous layer near the iron interface is seen to contain many of the inclusions which have been freed from the metal by the regression of the iron interface during the reaction. The porousness of this layer may be an indication that the inclusions act to initiate cracks and produce spalling of the sulfide layer on a microscale. By this process of the arrival of new inclusions at the interface and the removal of others by crack formation the effective interfacial area per unit area is essentially constant with time. Thus the parabolic growth law is obeyed but the measured rate is somewhat lower than the true rate.

The prolonged heat treatment involved in hydrogen firing partially spheroidized the elongated inclusions. This change in the average shape of the inclusion should decrease its effective blocking area at the interface and hence the measured rate of the reaction for such specimens should be greater than that for argon annealed specimens, where the inclusions retained their elongated forms (Figure 9). The data points (Figure 13) for the hydrogen treated specimens (circles) are slightly above those for the argon annealed specimens (squares).

Iron P, which contained a very large number of fine nonmetallics and some massive stringers, displayed a greatly reduced sulfidation rate. In Figure 13, these data plotted as filled circles are to be compared with the line defined by iron C when both were reacted at 800 C and 100 mm pressure.

Iron P is seen to show a reaction rate to be expected at a reaction temperature of 700 rather than 800 C. This marked reduction in the rate is directly attributable to the accumulation of inclusions at the Fe/FeS interface (Figure 10). Here the inclusions formed a nearly continuous layer at the interface greatly reducing the effective area for the transport of iron from the metal into the sulfide layer. The detached marker in Figure 10 indicates the location of an earlier longitudinal crack. The apparent soundness of the sulfide between this marker and the remaining iron implies that these inclusions do not themselves produce microspalling but that the detachment occurs by the formation of longitudinal cracks as described earlier. It also appears that the crack separates the sulfide layer from the accumulation of inclusion rather than progressing along the Fe/FeS interface. The detachment at the metal interface, as seen in the photomicrograph, probably occurred during cooling and not during the reaction period. Under these conditions the reaction rate must decrease with time more rapidly than predicted by the parabolic rate law since the effective interfacial

area has been reduced by the arrival of new inclusions at that plane and essentially none of this material is being removed from this site.

Calculations and Results

In order to compare the present data with those obtained by Hauffe and Rahmel[4] all reaction rate data have been converted to Wagner's rational rate constant.[10] This constant, K_r, is defined for the present reaction as the number of equivalents of iron transferred from the metal to the sulfide across a unit interfacial area (cm^2) in a unit time (second) when the sulfide layer is of unit thickness (cm). Since all of the data required for these calculations were not available some approximations and simplifying assumptions were required.

The sulfur content of the FeS at the Fe/FeS interface (S′)[(1)] was assumed constant (independent of temperature, sulfur vapor pressure in the reaction cell, and time of reaction). This is reasonable since (1) the low sulfur limit of the FeS field in the Fe-S phase diagram of Jensen[11] is invariant with temperature at 36.5 w/o[(2)], (2) there is strong evidence to indicate that the diffusion of iron through the scale is rate controlling and not the reactions at the interfaces, and (3) a free iron substrate remained for all experimental reaction times. The sulfur content of the sulfide at the $FeS/S_2(g)$ interface was obtained by a linear interpolation of Rosenqvist's[12] data. The data of Kelley[6] was used to convert Rosenqvist's (H_2S/H_2) pressures to sulfur vapor pressures. The average sulfur content ($\bar{S}$) was taken to be $(S' + S'')/2$. Data for the density of the sulfides were those given by Hägg and Sucksdorff[8] which compare very favorably with those reported more recently by Haraldsen.[13]

Linear Sulfur Gradient Across Scale Thickness

No direct evidence is available to support the simplifying assumption of a linear sulfur gradient across the thickness of the scale. However, a comparison of the experimentally observed ratio of the thickness of iron consumed to the thickness of sulfide produced with those calculated from the above data indicates that this assumption is reasonable. The experimental ratios, because of uncertainties in FeS thickness measurements, showed some scatter (enough to mask any trend with temperature) but centered about an average value of 0.36 cm^3 of iron to yield 1 cm^3 of sulfide. The calculated ratios are as follows: at 650 C—0.347, 700 C—0.352, 750 C—0.358, 800 C—0.363, 850 C—0.366, and 900 C—0.370 cm^3. Using data from these sources the present reaction rate constants (mm $Fe \cdot cm^{-2} \cdot hr^{-1/2}$) and those of Hauffe and Rahmel [(gm $S \cdot cm^{-2})^2 \cdot sec^{-1}$] were converted to Wagner's rational rate constant K_r (eq $Fe \cdot cm^{-2} \cdot sec^{-1}$). These have been tabulated in Tables 1 and 2.

(1) Single primed symbols refer to the metal-sulfide interface, double primed to the sulfide-gas interface, and barred symbols indicate average values.

(2) Rosenqvist's[12] data might be interpreted as showing a slight change in this composition with temperature—approximately 35.0 to 36.5 w/o S from 650 to 900 C.

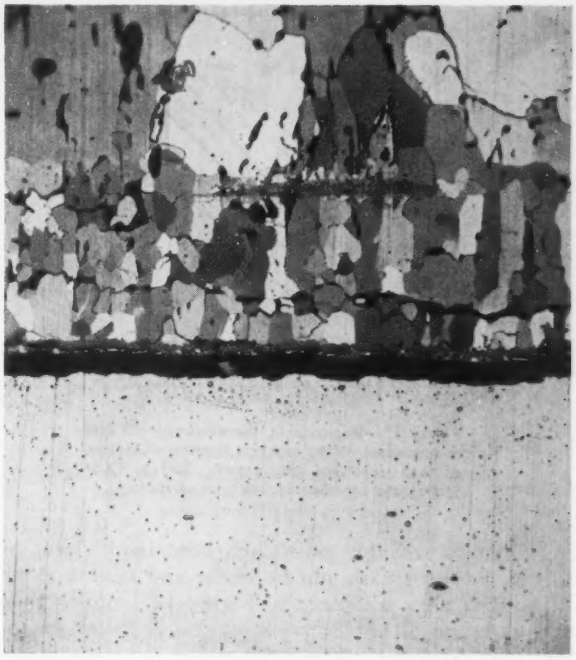

Figure 10—Fe/FeS interface and marker in Iron P specimen reacted at 800 C and 100 mm P_{S_2} for four hours showing almost continuous layer of non-metallic inclusions built up at interface. 125X, polarized light.

The growth of FeS on iron, as demonstrated by the retention of markers at the Fe/FeS interface, occurs by the migration of iron ions from the Fe/FeS interface to the FeS/S_2 interface where the iron ions react with sulfur from the gaseous atmosphere to form additional sulfide. The applicability of a parabolic growth law (Figure 2) indicates that diffusional flow through the scale is rate controlling. Hägg and Sucksdorff[8] have shown from density data that the sulfur rich lattice contains cation vacancies and not interstitial anions. Therefore the rate controlling process is taken to be a cation vacancy diffusion process.

On the basis of this mechanism the equation for the reaction may be written

$$4e^- + 2\,Fe^{++}(s) + S_2(g) = \underset{4h\,(e^-)}{2\,Fe^{++}(s)} + 2S^= + 2h\,(Fe^{++}) + \qquad (1)$$

where $h(Fe^{++})$ indicates a cation vacancy and $h(e^-)$ indicates an electron hole (or trivalent iron ion). Since the concentrations of e^-, Fe^{++} and $S^=$ are practically constant the mass action constant may be written

$$K = \frac{[h(Fe^{++})]^2\,[h(e^-)]^4}{P_{S_2}} \qquad (2)$$

From equation (1), $2[h(Fe^{++})] = [h(e^-)]$, hence

$$[h(Fe^{++})] = K'(P_{S_2})^{1/6} \qquad (3)$$

Thus the cation vacancy concentration in the sulfide lattice at constant temperature is proportional to the 1/6 power of the sulfur vapor pressure. The reaction rate constant, K_r, should be proportional to the concentration of vacancies if the vacancies do

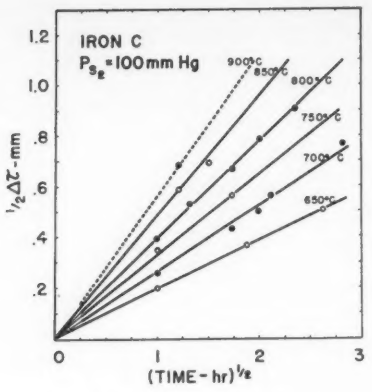

Figure 11—Decrease in the thickness of iron C specimens during reaction time as a function of the reaction temperature. Sulfur vapor pressure constant at 100 mm of mercury.

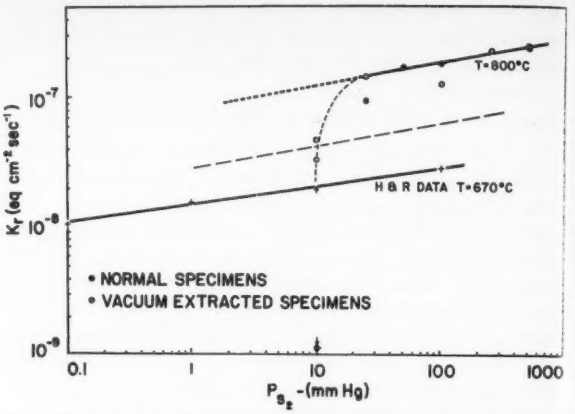

Figure 12—The effect of sulfur vapor pressure (P_{S_2}) on the rate constant (K_r) for iron C. Temperature = 800 C.

not interact strongly with each other and a plot of log K_r vs log P_{S_2} should be linear and have a slope of 1/6. The data presented in Figure 12 confirms that the variation of log K_r with log P_{S_2} is linear (from 500 mm P_{S_2} down to the point where the experimental conditions failed—at approximately 10 mm P_{S_2}) and the slope of this line is approximately 1/6.

Hauffe and Rahmel[4] observed the same behavior at 670 C, and because of the nature of their experimental conditions they were able to extend their measurements to very low pressures. These data are also shown in Figure 12.

Since the growth of the sulfide layer has been shown to be controlled by the vacancy diffusional flow of iron ions alone, it is possible, through Wagner's analysis,[10] to evaluate an average self-diffusion coefficient for iron in the sulfide lattice. Because of the lack of detailed knowledge of the sulfur concentration gradient existing in the scale, and hence of the concentration of cation vacancies, the values of this diffusion coefficient must be considered only as a very crude approximation. Direct measurements of the self-diffusion coefficients in FeS by the use of radioactive isotopes are now being attempted in this laboratory.

For the present calculations the relation proposed by Wagner is the following:

$$K_r = C_{eq} \int_{a'_s}^{a''_s} \left[\frac{Z_{Fe}}{|Z_S|} D^*_{Fe} + D^*_S \right] d \ln a_S \qquad (4)$$

Here D^*_S, the self diffusion coefficient for the anion, may be considered negligibly small; Z_S, the valence of the anion, is taken as two; $C_{eq} = C_{Fe} Z_{Fe} = C_S |Z_S|$ where the concentrations C_{Fe} and C_S are in moles/cm^3; and K_r is the rational rate constant as defined previously. Thus equation (4) becomes

$$K_r = \bar{C}_S \int_{a'_s}^{a''_s} Z_{Fe} \bar{D}^*_{Fe} \, d \ln a_S \qquad (5)$$

$$K_r = 2.303 \bar{C}_S \bar{D}^*_{Fe} [Z''_{Fe} \log P''_{S_2} - Z'_{Fe} \log P'_{S_2}] \qquad (6)$$

where Z''_{Fe} is the valence of iron at the gas interface and Z'_{Fe} is the valence at the metal interface, P'_{S_2} is the decomposition pressure of FeS, and P''_{S_2} is the sulfur vapor in the reaction cell. Thus at P''_{S_2} 100 mm Hg.

$$K_r = 4.606 \bar{C}_S \bar{D}^*_{Fe} [Z''_{Fe} - \log P'_{S_2}] \qquad (7)$$

Rational rate constants and the values of $\bar{D}^*_{Fe}$ calculated from these are listed in Table 1 and plotted on a logarithmic scale against the reciprocal of the experimental temperature (degrees K) in Figure 14. The data of Hauffe and Rahmel, converted to K_r, also are given in this table. The activation energy indicated for this approximation of $\bar{D}^*_{Fe}$ is 20,600 cal/mole.

Discussion

Comparison of the present rate data with those of Hauffe and Rahmel show a consistent disagreement. In Figures 12 and 14 the dash lines represent the present data adjusted to the conditions of Hauffe and Rahmel's experiments. They indicate rate constants almost twice as large as those determined in the earlier work. Possible contributions to this difference are discussed below.

Hauffe and Rahmel characterized their specimen material as electrolytic iron but gave no analysis. In view of the results obtained here on Armco iron, it is unlikely that the difference in the compositions of the materials used in these studies could account for the difference in the results, although some contribution might come from this source.

The geometry of the specimens employed and the methods used in determining the rate of reaction may account for more of this difference. Hauffe and Rahmel used specimens 0.20 mm in thickness (other dimensions not reported) and measured the total weight gain of the specimen. The data were reported only as a derived plot (weight of sulfur per cm^2 of interface)^2 versus time and the method of evaluating the total interfacial area was not mentioned. The curves (wt S•cm^{-2})^2 vs t, indicated a parabolic behavior throughout most of the reaction time. The last data points on the curves at 670 C indicated a

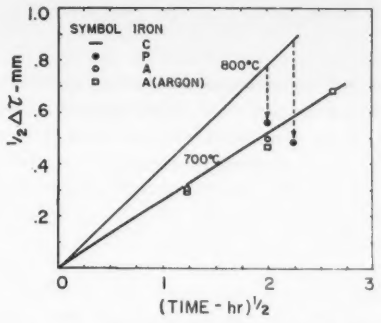

Figure 13—The effect of non-metallic inclusions on the rate of the reaction. Sulfur vapor pressure constant at 100 mm of mercury.

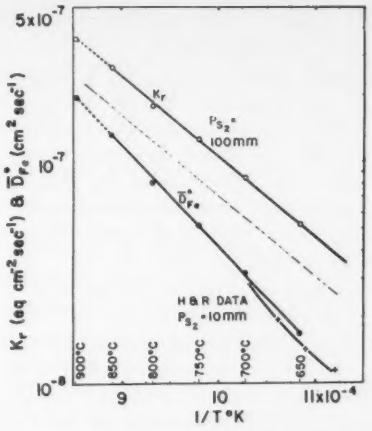

Figure 14—The effect of reaction temperature on iron ion self-diffusion coefficient and the rate constant (K_r) for iron C. Sulfur vapor pressure = 100 mm of mercury.

wt gain/cm² of 4.8×10^{-2} gm/cm² corresponding to a sulfide layer approximately 0.26 mm in thickness—requiring the consumption of 0.09 mm of iron. Since this reaction occurs on both faces of the specimen the thickness of the remaining iron should be 0.02 mm. If the calculations of weight gain per unit area were based on the original total surface area (i.e., assuming that edge and corner effects were negligible and that the iron core retained its original length and width—decreasing only in thickness), then the derived reaction rates should be low. Deviations from parabolic growth are to be expected also.

These authors also indicated that when a specimen of iron which had completely reacted to FeS was broken open a central void was observed. This, they concluded, was a manifestation of the vacancy migration of iron ions through the sulfide. Hence they observed a detachment of the sulfide. No study of the microstructure of these specimens was indicated, but the detachment and the cracking present in their specimens may have been more severe than that shown in the photomicrographs included in this paper. In this present study only a few specimens were inadvertently converted entirely to sulfide. Polished sections of these revealed some central porosity but no extensive cavity.

Uncertainty of Temperature

Difficulties in determining the true specimen temperature may, in part, account for the lower reaction rates and the curvature of the log K_r vs 1/T line of the Hauffe and Rahmel data shown in Figure 14. These investigators were forced to accept a temperature uncertainty of ±4 degrees C in the upper furnace (iron specimen) and ±2 degrees C in the lower furnace (sulfur reservoir) of their vertical tandem furnace arrangement. In the preliminary work for the present studies the possibility of using similar furnaces was investigated. Experiments showed that it was extremely difficult to obtain the desired temperature distribution in vertical furnaces and that the temperature distribution was not reproducible. The gradual curvature of Hauffe and Rahmel's data shown in Figure 14, may be a consequence of the overheating of the specimens due to the heat of formation of FeS which would be most important at the higher temperatures. These specimens were very thin (0.20 mm) and had a large surface to volume ratio. These K_r values calculated from Hauffe and Rahmel's data were subject to the same averaging in conversion as the present results.

The ability of these investigators to obtain rate data at low sulfur pressures, unobtainable in the current studies, may be the result of the difference in the geometry of their reaction vessel from that employed here. Their reaction cell appears to have been much larger and thus able to hold small amounts of gaseous impurities without seriously interfering with the sulfur vapor concentration.

No other comparison of rate data for the growth of FeS on iron is possible since most of the other investigations reported in the literature are essentially alloy evaluation studies.[1,2,14] In these experiments various sulfur bearing atmospheres were employed but the experimental conditions rarely yielded definitive rate data.

Rate of Growth of FeO and FeS

A comparison of the rate of growth of FeO and FeS is of interest. The structure of these two phases are much more similar than is indicated by the normal representation of their ionic arrangement. FeS is a NiAs type lattice, while FeO is NaCl type. In FeS the structure can be built up of close packed planes of atoms characterized by the form (a B a C a B a C - - -), where "a" represents a plane of iron ions and "B" and "C" planes of sulfide ions. The arrangement of the sulfide ions corresponds to hexagonal close packing. This is very nearly the case in this phase—the sulfide ion packing is only slightly distended by the insertion of the planes of iron ions. These iron ions occupy octahedral holes between the anion planes. The FeO lattice may be viewed as two interpenetrating face centered cubic lattices one containing the anions, the other, the cations. Each of these is characterized by a close packed plane sequence (ABC ABC . . . . .). Thus in the oxide the anions are

very nearly close packed and the cations fill interstices between these planes.

Both of these phases exist over a range of compositions—the composition change being effected by the removal of iron ions from the lattices with electrical neutrality preserved by converting an appropriate number of ferrous ions to the ferric state. Since the growth of each of these phases occurs by vacancy diffusional flow of the cations a comparison of rates is plausible.

Using the data of Himmel, Mehl, and Birchenall[15] for wüstite growth at 800 C, the rational rate constants for FeO and FeS are respectively 0.25×10^{-8} and 18×10^{-8} eq cm^2 sec^{-1}. From measurements of iron self-diffusion in these phases the activation energies are given as 29,700 cal/mole for FeO containing 0.907 moles of Fe per mole of 0 and 20,600 cal/mole for "FeS"—a very rough estimate from samples in which the mole ratio of iron to sulfur varied from one at the Fe/FeS interface to 0.84 at the $FeS/S_2(g)$ interface. Thus the higher growth rate of the sulfide may be associated with the larger size of the sulfur ion with respect to the iron ion and the greater concentration of vacancies permissible in the iron ion sites.

The interpretation given these experiments is not in agreement with a recent result reported by Pfeiffer and Ilschner.[16] They sulfidized iron in normal sulfur vapor for 10 minutes at 670 C then for 120 minutes in sulfur vapor to which radioactive sulfur had been added. Rates were not measured on these samples nor were the cross sections examined metallographically. The sulfide scale was removed mechanically from the substrate, and the radioactivity was measured from both sides of the scale and found to be the same. If the whole scale was removed and not just the outer part (note the porous plane of mechanical weakness in Figure 6) and if no connection existed during the growth period between the interior of the sulfide and the vapor phase (note the corner effect in Figure 2), then the result clearly implies that sulfide ions diffuse about as rapidly as iron ions in the sulfide lattice during growth. Preliminary iron self-diffusion measurements in this laboratory confirm the unusually high mobility of the iron ions, and the marker evidence presented here supports a much lower mobility for sulfide ions. In view of the much greater size of the latter and the brittleness of the crystals it is difficult to believe that the radioactive sulfur experiment of Pfeiffer and Ilschner is as simple in detail as it has been assumed to be.

Summary

1. Inert chromium oxide markers retained at the Fe/FeS interface during the growth of the sulfide layer show that the iron ion is much more moble than the sulfide ion in the FeS lattice.

2. The accumulation of nonmetallic inclusions during the growth of FeS on the Armco iron and iron P confirms the marker movement data.

3. Sulfidation of a Fe:FeS eutectic specimen showed conclusively that the diffusion of the iron ion is largely responsible for the growth of the sulfide layer.

4. A detailed metallographic study of these specimens has revealed that inert markers may be displaced from the interface without the necessity of a diffusional flow of anions past the markers. In these studies some markers have been found deeply imbedded in the sulfide layer while others remained at their original site, i.e., the surface of the iron specimen. Markers have been shown to become detached from the interface by (1) crack formation resulting from the stresses developed in the growing scale and (2) an undercutting action due to obstruction of the diffusional flow of iron ions in the growth process. These observations indicate the importance of careful metallography and a detailed study of all aspects of the growth process in evaluating marker movement data.

5. Because of the stress system developed in the sulfide layer, longitudinal cracks frequently formed along one side of the specimen, detaching the sulfide from the iron at this interface. The equality of the thickness of the sulfide layer formed in cracked and uncracked regions indicated that this detachment did not alter the rate of the reaction. The mechanism for the continued growth that was observed has not been investigated in detail but is believed to involve thermal dissociation of the sulfide to provide sulfur vapor for further reaction inside the crack.

6. Temperature measurements during the initial reaction period have shown that the high initial reaction rate and the high heat of formation of FeS may temporarily raise the specimen temperature significantly. Because of the magnitude of this temperature rise, experiments above 900 C were impractical.

7. Measurements of the volume of iron converted to sulfide as a function of reaction time conform to a parabolic growth law. Thus the growth of FeS is controlled by the diffusional flow of iron and not by interface reactions.

8. Rational rate constants have been determined for the growth of FeS at a sulfur vapor pressure of 100 mm of mercury over the temperature range 650 to 900 C.

9. An average self-diffusion coefficient for iron in the FeS lattice has been calculated. Because of the approximations employed, the values of this diffusion coefficient are not highly accurate. These calculations indicate an activation energy of the order of 20,600 cal/mole for the vacancy diffusion of iron ions in the sulfide.

10. The effect of sulfur pressure on the rate of the reaction is in agreement with the variation predicted by Wagner's theory of scaling. Because of design factors in the equipment it was not found possible to obtain reliable data at sulfur vapor pressures below 25 mm of mercury.

11. These results have been compared with the data obtained by Hauffe and Rahmel[4] for the growth rates of FeS on iron. The rates observed in the present work are somewhat higher than those previously reported.

Acknowledgment

This research was supported by the United States Air Force through the Air Force Office of Scientific Research of the Air Research and Development Command under Contract No. AF 18(600)-967.

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Any discussions of this article not published above will appear in the December, 1957 issue.

A Laboratory Study of N-Oleoyl Sarcosine As a Rust Inhibitor in Some Petroleum Products*

By ROBERT M. PINES and JOHN D. SPIVACK

Introduction

N-ACYLAMINO ACIDS previously have been shown to have effective rust inhibiting properties in mineral lubricating oil solution.[1] Among the most effective members of the compounds studied are the fatty acyl derivatives of sarcosine, $HNCH_2COOH$ (with CH_3 on the N).

N-Oleoyl sarcosine (I) is readily

$$CH_3(CH_2)_7CH=CH(CH_2)_7CONCH_2COOH$$
(with CH_3 on the N)

I (N-oleoyl sarcosine)

adaptable for formulation in petroleum products because it is (a) liquid at room temperatures, (b) readily soluble in hydrocarbons as well as syntheic lubricants, (c) ashless upon ignition, in addition to its performance properties under a variety of rust-inducing conditions. The physical properties of N-oleoyl sarcosine are summarized in Table 1.

Baker et al[2] have pointed out the importance of selecting specialized test methods simulating service conditions for rust inhibitor evaluation, since the effectiveness of polar organic compounds is not directly deducible from classical adsorption studies. Liquid solutions of N-oleoyl sarcosine were subjected to test methods adopted by government and industry for use in evaluating the rust inhibiting properties of gasoline, kerosene and other distillate fuels, lubricants, hydraulic, circulating, instrument and preservative oils. Auxiliary tests also were applied to determine the effect the addition of N-oleoyl sarcosine might have on other properties of some of these products.

N-Oleoyl Sarcosine in Gasoline

Hydrocarbon soluble inhibitors have mitigated direct and indirect losses due to internal corrosion in product pipelines[3] and marine tankers[4] carrying refined petroleum products such as gasoline and kerosene.

Laboratory studies of the rust preventive properties of N-oleoyl sarcosine in a straight run gasoline containing no additives or dye were run under a variety of conditions using standard tests as well as special tests devised in this laboratory.

Dynamic Immersion Test (Method A.S.T.M. D665-54[5] Modified)

The test was modified to run at 80 F for three hours with synthetic sea water[5] and was designed to evaluate the rust preventive properties of inhibited gasoline when both the aqueous and hydrocarbon phases are intimately stirred and both in contact with the entire immersed portion of the SAE 1020 steel test cylinder. The same test also was run on inhibited gasoline which had been subjected to a water extraction with ten percent by volume of distilled water. Data are shown in Table 2.

Static Immersion Test

This test[6] which is run under essentially quiescent conditions, simulates those found in marine tankers during the cargo run and in storage tanks.

★ Submitted for publication November 5, 1956. This article is based on a paper presented at the Atlantic City meeting of American Chemical Society, September, 1956.

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Abstract

Laboratory studies indicate that N-oleoyl sarcosine (I) is an effective oil soluble rust inhibitor in solution in gasoline, mineral oil and silicone lubricants.

Rusting tests used included: Turbine Oil Rusting Test (ASTM D665-54), a Static Immersion Test, Humidity Cabinet Test (JAN H-792), Static Water Drop Test (MIL-L-17353 Bu. Ord.). In addition, a number of auxiliary tests were run to indicate the effect of (I) on other properties of the test liquid.

Hydrocarbon solutions of N-oleoyl sarcosine are particularly effective in preventing rusting in the presence of synthetic sea water and in resisting water extraction of the inhibitor. 5.8.2

A 6½ x 1 x 1/32 inch SAE 1010 cold-rolled steel strip cleaned and finished with 5/0 sandpaper is partially immersed in 100 ml of ASTM synthetic sea water and 750 ml of gasoline contained in one quart jar. The system is thoroughly shaken for one minute and then set aside in the dark for three weeks at room temperature. The specimen is then removed, examined and rated by the system described in Table 2. Table 3 indicates the unusual effectiveness of N-oleoyl sarcosine in preventing corrosion of the steel exposed to the aqueous phase.

The elimination of rusting of test specimens immersed in gasoline solutions of N-oleoyl sarcosine is shown in Figure 1. Increase of the concentration of N-oleoyl sarcosine from 0.005 percent to 0.01 percent practically eliminates corrosion of that portion of the test specimens exposed to the vapor phase.

Reverse Cycling Test

This test was devised to simulate conditions in a marine tanker through one complete cycle in which the bulkheads are filled first with refined cargo, then are either ballasted with sea water or are maintained empty for the return trip. Tests similar in principle, if not in detail, have been used by others.[7]

Tared, sandblasted specimens of SAE 1010 cold-rolled steel, 1 x 3 x 1/32 inch in size are rusted by immersion in synthetic sea water for four days. They are air dried for one day, reimmersed in synthetic sea water for one day and finally air dried for one day more. This produces an adherent relatively uniform rust coating. The specimens are wetted in synthetic sea water and totally immersed in the test gasoline for one week at room temperature. They are then removed and half the number of specimens are totally immersed in synthetic sea water for one week more. The rest of the steel strips are suspended from a rack which is rotated slowly over synthetic sea water contained in a covered dessicator jar. Finely dispersed air is blown through the sea water so that a spray collects on the specimens and drips from them as they rotate. After one week at room temperature all the specimens are removed, brushed well and cleaned in inhibited acid. They are reweighed and percent protection calculated from the following equation.

$$\text{Percent Protection} = 100 \times \frac{\text{Wt. loss uninhibited} - \text{Wt. loss inhibited specimen}}{\text{Wt. loss of unhibited specimen}}$$

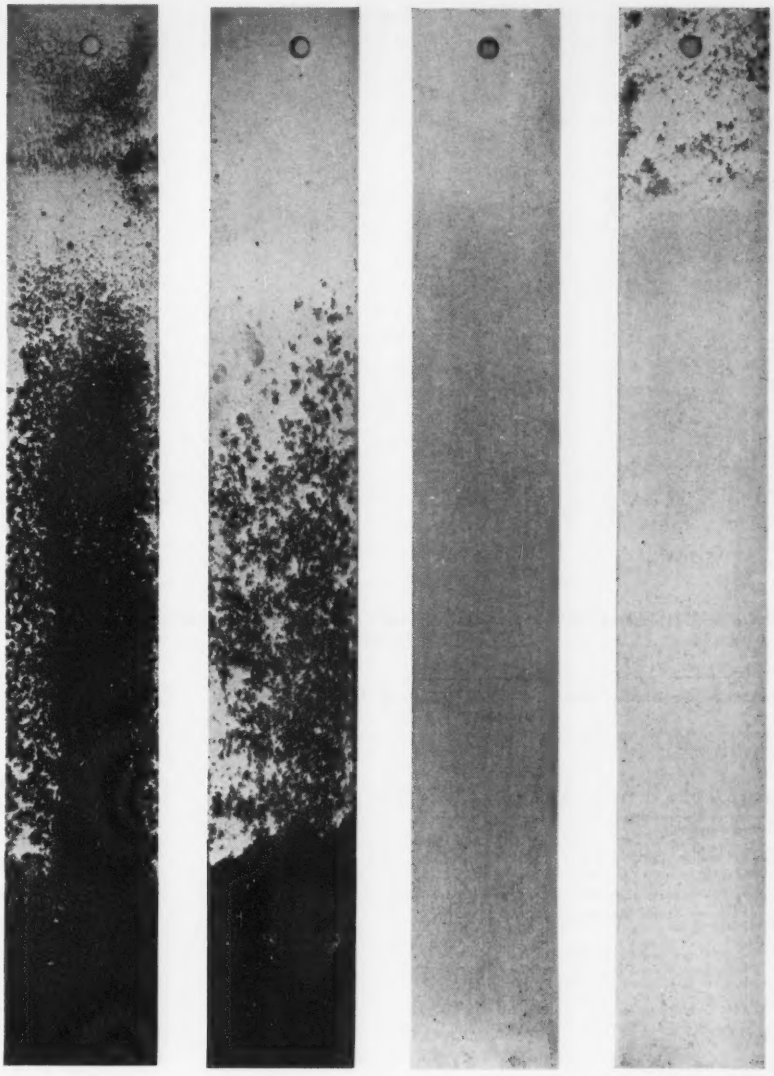

Figure 1—SAE 1010 cold rolled steel strips after immersion in inhibited or uninhibited gasoline.

TABLE 1—Physical Properties of N-Oleoyl Sarcosine

Setting Point	32 F
Viscosity at 100 F	1180 SUS (255 CPS)
Color (ASTM)	3
Residue upon Ignition	None
Specific Gravity	0.948
Molecular Weight	340–350
Solubility at Room Temperature	
In Paraffin Oil[1]	Infinite
In Kerosene	Infinite
In Stoddard Solvent	130 gms/100 gms.
In Water	Insoluble
Stability of 1% Solution in Paraffin Oil[1] at 12 F for 8 months	Clear

[1] Solvent-extracted mineral oil of 182 SUS at 100 F—Regal Oil B—Texas Co.

The test results (Table 4) emphasize the severity of the sea water spray test compared to the sea water immersion test, and the superiority of N-oleoyl sarcosine in both systems compared to the commercial inhibitor.

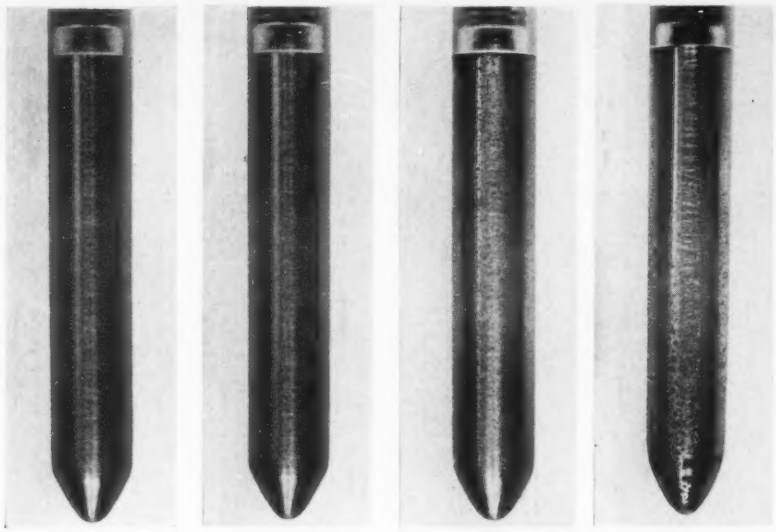

Figure 2—Test pieces after subjection to inhibited or uninhibited turbine oil (Test—ASTM D665-54 Procedure B).

TABLE 2—Results of Modified ASTM D665-54 Test, Inhibitors in Gasoline[1]

Inhibitor	Concentration Weight Percent	RATING[2] Before Extraction	RATING[2] After Extraction
N-oleoyl sarcosine	0.005	1	1
	0.0025	4	
Commercial Inhibitor S	0.005	2	5
None	.....	7	

[1] Socony-Mobil Marine, no additives or dye
[2] Rating System
1. Free of rust
2. Trace of rust, few spots
3. Less than 5 percent of surface rusted
4. 5–50 percent of surface rusted
5. 50–99 percent of surface rusted
6. Surface covered by light rust
7. Surface covered by heavy rust

TABLE 3—Results of Static Immersion Test, Inhibitors in Gasoline[1]

Inhibitor	Concentration Wt. Percent	RATING[2] OF SAE 1010 STEEL IMMERSED IN Sea Water	Gasoline	Vapor
N-oleoyl sarcosine	0.01	1	1	1
	0.005	1	1	4
	0.0025	1	7	4
	0.001	5	6	5
Commericial Inhibitor S	0.01	7	4	1
None	.....	7	6	6

[1] Socony-Mobil Marine, no additives or dye.
[2] See Table 2 for rating system.

TABLE 4—Reverse Cycling Test, Inhibitors in Gasoline[1]

Inhibitor	Concentration Weight Percent	PERCENT PROTECTION Specimens in Sea Water	Specimens in Spray
N-oleoyl sarcosine	0.0025	44	6
	0.0200	71	37
Commercial Inhibitor S	0.0025	13	0
	0.0200	19	6

[1] Socony-Mobil Marine, no additives or dye.

The dynamic, static and cycling corrosion tests all demonstrate the rust preventive properties of gasoline solutions of N-oleoyl sarcosine under a variety of corrosive conditions. Additional tests were run to determine the effect of the inhibitor on some other properties of engine fuels.

Solubility of Lead Salt of N-Oleoyl Sarcosine

The lead salt of N-oleoyl sarcosine was prepared by double decomposition of the sodium salt and lead acetate and was found to be soluble at room temperature in gasoline in the concentration range tested (up to 0.5 percent).

Water Tolerance of Aircraft Fuels (ASTM D1094-53)

This method[5] provides a procedure for determining the solubility of water in aviation gas and jet turbine fuels and involves shaking a sample of fuel with water at room temperatures and measuring the volume change of the aqueous layer. N-oleoyl sarcosine apparently has no adverse effect on the solubility of water in gasoline and affects the emulsion properties only slightly in the concentrations tested (up to 0.01 percent).

Oxidation Stability of Gasoline (ASTM D525-55).

Rust inhibitors have frequently acted as pro-oxidants in the catalyzed oxidation of hydrocarbon liquids. To study the effect of N-oleoyl sarcosine, the inhibitor was dissolved in a less stable gasoline containing rust inhibitor, antioxidant, tetraethyllead and dye. The data indicate that N-oleoyl sarcosine had a marked stabilizing effect on the test gasoline (0.0025 percent increased oxygen stability from 6 to 7½ hours while 0.005 percent increased stability to nine hours.)

Copper Corrosion by Petroleum Products (ASTM D130-55T)

N-Oleoyl sarcosine in gasoline at a concentration of 0.005 percent did not corrode or tarnish copper in the ASTM test.

Lubricants, Hydraulic, Circulating, Instrument and Preservative Oils

Oil-soluble rust inhibitors combining rust prevention properties, chemical and thermal stability, and water demulsibility are required for the lubrication and protection of closely engineered mechanisms found in steam and gas turbines, engines, servomechanisms, instruments, etc. The test methods employed for evaluating the rust preventive efficiency

of N-oleoyl sarcosine in petroleum and synthetic liquids designed for such systems were the following:

1. Static Water Drop Test (MIL-L-17353 [Bu. Ord.])
2. Turbine Oil Rusting Test (ASTM D665-54, Proc. B)
3. Humidity Cabinet Test (JAN H-792).

The Static Water Drop Test has been noted[2] as being more severe than the ASTM Turbine Oil Rusting Test but less drastic in causing rusting than either the JAN-H-792 Humidity Cabinet Test[8] or the Naval Research Laboratory Fog Cabinet Test.[9]

Static Water Drop Test (MIL-L-17353 [Bu. Ord.]).

The mineral oil used in this and the following tests, unless otherwise noted, is a non-additive solvent-extracted turbine grade lubricating oil of 180 SUS at 100 F sold by the Texas Co. as Regal Oil B. N-Oleoyl sarcosine is miscible with this oil in all proportions at room temperature. Similarly, one percent oil solutions of N-oleoyl sarcosine kept at 12 F (—7 C) have remained stable and clear for a period of more than eight months.

The Static Water Drop Test involves observing rust formation at 140 F in a 1.75 inches triangular cold-rolled steel specimen immersed in oil, when a 0.2 milliliter drop of distilled water is inserted in the centrally located dimple of the test-piece.

Inhibitor efficiency in this test was determined in two ways depending on the nature of the aqueous phase:

(a) When distilled water was used, a series of varying concentration runs (0.001 to 0.20 percent) were made and the minimum effective concentration estimated, and

(b) When synthetic sea water was used, the additive was tested at 0.05 percent and the time to rust noted.

The results indicate that N-oleoyl sarcosine is among the most effective additives tested by this method and is particularly effective when synthetic sea-water is used as the corrosive agent (Table 5). N-Oleoyl sarcosine is also very effective in solution in silicone oil. The rust preventive action of a silicone solution of N-oleoyl sarcosine was not adversely affected by heating to 450 F for eight hours prior to testing.

Turbine Oil Rust Test (ASTM D665-54, Procedure B)

This method involves stirring a mixture of 300 ml of oil under test with 30 ml of synthetic sea water at a temperature of 140 F with a cylindrical steel specimen completely immersed therein. Results (Table 6) indicate the greater protection against rusting under dynamic flow conditions provided by N-oleoyl sarcosine compared to two other commercially available inhibitors tested.

The elimination of rusting in this test by lubricating oil solutions of N-oleoyl sarcosine compared to the rusting permitted by commercial inhibitors L and P is shown in Figure 2.

Humidity Cabinet Test (JAN-H-792)

Following the system of panel preparation described in MIL-L-3150 Lubricating Oil Preservative, Medium, mineral oil solutions of N-oleoyl sarcosine afforded substantial protection. The test data (Table 7) indicate that N-oleoyl sarcosine is more effective than a typical commercial petroleum sulfonate at concentrations above one percent.

In several instances the properties of N-oleoyl sarcosine have been improved by its use in conjunction with organic amines. For example, when combined in equal weight concentration with a substituted 2 heptadecenyl imidazoline (Amine O)—Geigy Chemical Corporation) incorporated into Regal Oil B, and tested in the JAN H-792 Humidity Cabinet, the protection afforded is increased some four-fold.

Mineral oil solutions of N-oleoyl sarcosine were also tested to determine the effect of these materials on the emulsification and oxidation properties of oil solutions. This is particularly important in petroleum products where the formation of a stable water emulsion and too great a reduction of the efficacy of the antioxidants is undesirable. The oxidation test used is a modification developed by the Aluminum Com-

TABLE 5—Static Water Drop Test—140 F

Inhibitor	Base Oil	Distilled Water Min. Effect[1] Conc.— (Wt. Percent)	Synthetic Sea Water Inhibitor Conc.-0.05 Percent Time to Rust (Hrs.)
N-oleoyl sarcosine	Mineral[2]	0.005	20
Alkenyl succinic[4] acid	Mineral[2]	0.005	1
Mixed mono- and di-dodecylphosphates[4]	Mineral[2]	0.025	1
N-oleoyl sarcosine	Silicone[3]	0.0125[5]	..
		0.0125	..

[1] Estimated from rusting tests at varying concentrations.
[2] Solvent-extracted turbine grade oil, Regal Oil B., Texas Co.
[3] Versilube F50, General Electric Co.
[4] Commercial samples.
[5] After heating for 8 hours at 450 F (232 C).

TABLE 6—Turbine Oil Rusting Test (ASTM D665-54) Procedure B Synthetic Sea Water—140 F, Mineral Oil[1]

Inhibitor	Concentration Weight Percent	Observations
N-oleoyl sarcosine	0.0125	Completely free of rust or staining
	0.025	Completely free of rust or staining
Commercial inhibitor	0.025	Light rusting
Commercial inhibitor	0.025	Moderate rusting

[1] Solvent extracted turbine grade oil, Regal Oil B., Texas Co.

TABLE 7—Mineral Oil Solutions of N-Oleoyl Sarcosine in the Humidity Cabinet Test (JAN H-792)

Inhibitor	Conc. (Active Basis) Weight Percent	Time to Failure (Hours)
N-oleoyl sarcosine	0.25	80
	0.50	130
	0.75	144
	1.00	175
	2.00	472
	3.00	590
	4.00	400
	5.00	590
Amine O	0.5	72
N-oleoyl sarcosine 1 part	0.5	960
plus Amine O 1 part	0.5	
Sodium petroleum sulfonate[1]	1.00	300
	2.00	200
	3.00	300
	4.00	200
	5.00	140
None	....	<20

[1] Commercial oil soluble petroleum sulfonate, average molecular weight —490.

pany of America[10] of the method described by Davis, et al[11] involving the measurement of the adsorption of oxygen by 13 milliliters of test oil at 115 C, in the presence of copper catalyst wire. The test results indicate that N-oleoyl sarcosine does not unduly affect the emulsification characteristics of the base oil, but does reduce the effect of the antioxidant, 2,6 ditertiary butyl paracresol, by reducing oxygen stability from 105 hours to 60 hours. The untreated mineral oil had an oxygen stability of 12 hours.

Other Rust-Inhibiting Applications for N-Oleoyl Sarcosine

Baroid Sales, Division of National Lead Co., has conducted a series of tests which demonstrate the effectiveness of N-oleoyl sarcosine as a corrosion inhibitor for bentonite greases.[12] Preliminary testing at the Research Laboratories of Geigy Chemical Corporation has shown the corrosion inhibiting effectiveness of N-oleoyl sarcosine in cutting oil formulations.

Conclusions

N-oleoyl sarcosine has been subjected to a variety of rusting tests designed to indicate its suitability for use as an oil-soluble rust preventive in gasoline and other distillate fuels, lubricating, circulating, hydraulic instrument, preservative oils and bentonite greases. N-Oleoyl sarcosine provides effective rust inhibition under static and dynamic type immersion tests as well as in the presence of humid corrosive atmospheres.

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Any discussions of this article not published above will appear in the December, 1957 issue.

TECHNICAL COMMITTEE ACTIVITIES

Report Shows Progress Made on Zero Resistance Ammeter for Electrolysis Tests

"A Zero Resistance Ammeter for Use in Electrolysis Testing Work" is the title of a report recently submitted to Unit Committee T-4B on Corrosion of Cable Sheaths by Task Group T-4B-3B on Tests and Surveys. The report was prepared by Work Group T-4B-3B, D. R. Werner, American Tel. & Tel. Company, Kansas City, Missouri, chairman.

The report was first submitted to T-4B at the Twelfth Annual Conference in New York in March 1956 in the form of a memorandum which covered the combined contribution of a number of different organizations interested in the development of a zero resistance ammeter for the measurement of current flow from lead sheath cables in manholes.

In the original presentation the work group indicated the method appeared to give inaccuracy of measurement within about five to ten milliamperes. Thermal electric potentials developed in the galvanometer circuit appeared to be the prime source of error. These were especially troublesome for the measurement of currents of less than about 50 milliamperes when only 18 inches of cable sheaths about 2½ inches in diameter was available.

Cases where the heat of the operator's fingers on the galvanometer push button caused thermal electric potentials to be set up at the push button contacted were reported to the group. This could be over-come by providing thermal insulation.

Heat generated within the set by the current controlling resistors or sun shining on the set might cause even greater changes. Thermal insulation placed around the galvanometer thermal potentials has been used to minimize the generation of currents in the galvanometer circuit.

Since the report was presented at New York, a working model has been built and given a field trial. The field trial indicated a change should be made in the circuit arrangement for testing the resistance of the galvanometer test leads to make it more convenient to calibrate the instrument. These changes are incorporated in the report recently circulated to the committee for letter ballot.

FORMAL PAINTING PROGRAMS QUESTIONNAIRE

A painting program questionnaire has been distributed to engineers in several hundred plants covering the United States by the Painting Program Task Group of Unit Committee T-6D on Industrial Maintenance Painting. The questionnaire is designed to develop information on the extent to which formal painting programs are used in the process industry.

The task group hopes to give a report on replies to the questionnaire at the T-6D meeting in Oklahoma City on October 2.

Pipe-Type Cable Protection Questionnaire Circulated

A questionnaire on protection of pipe-type cables has been distributed by Task Group T-4B-4. The results of a previous survey of corrosion protective practices on pipe type cables was published in CORROSION, Vol. 10, No. 9, 299-313 (September). The current questionnaire is designed to bring up to date information on coatings, cathodic protection and grounding.

Questions on coatings include type of coating used, reinforcing and shielding thickness, deterioration and minimum acceptable coating resistance in initial test of buried pipe.

Questions on grounding are designed to establish how the pipe is grounded at the source end, if it is connected in route, grounded at remote source end and if any sections of pipe are operated with insulating joints in pipe.

Questions on cathodic protection will establish type of cathodic protection used and the value of pipe potentials in soil.

Results of the questionnaire will be presented to Unit Committee T-4B and ultimately submitted for publication in CORROSION.

Burton to Head T-5A

Walter H. Burton, General Chemical Division, Allied Chemical & Dye Corp., Camden, New Jersey, has been elected vice-chairman of Unit Committee T-5A, Chemical Manufacturing Industry.

Mr. Burton has long been active in T-5A-5 on Nitric Acid, and T-5A-6 on Hydrofluoric Acid. He is a member of ASTM C-3 on chemical resistant mortars and a member of the High Alloy Steel Committee of the Welding Research Council.

Technical Articles Scheduled for November Publication

Some Experience With Sodium Silicate as a Corrosion Inhibitor in Industrial Cooling Waters by J. S. Beecher, J. W. Wood and P. S. Laurence

Fundamentals of Electrode Processes in Corrosion by Milton Stern

Corrosion and Adsorption Studies Using Sulfonate Inhibitors by A. H. Roebuck

Factors Other Than Mineral Content Which Influence the Corrosiveness of Cooling Water by E. H. Hurst

Some Concepts of Experimental Design by John D. Hromi

Inhibiting a Cooling Water Tower System by F. L. Whitney, Jr.

Non-Chemical Factors Affecting Inhibitor Selection and Performance in Air Conditioning Cooling Waters by Sidney Sussman

An Evaluation of Inhibitors for Corrosion Prevention in an Engine Cooling System by Leonard C. Rowe

Current Requirements for Cathodic Protection of Oil Well Casing by E. W. Haycock

A Thermochemical Study of Some Additives to Reduce Residual Fuel Ash Corrosion by W. E. Young and A. E. Hershey

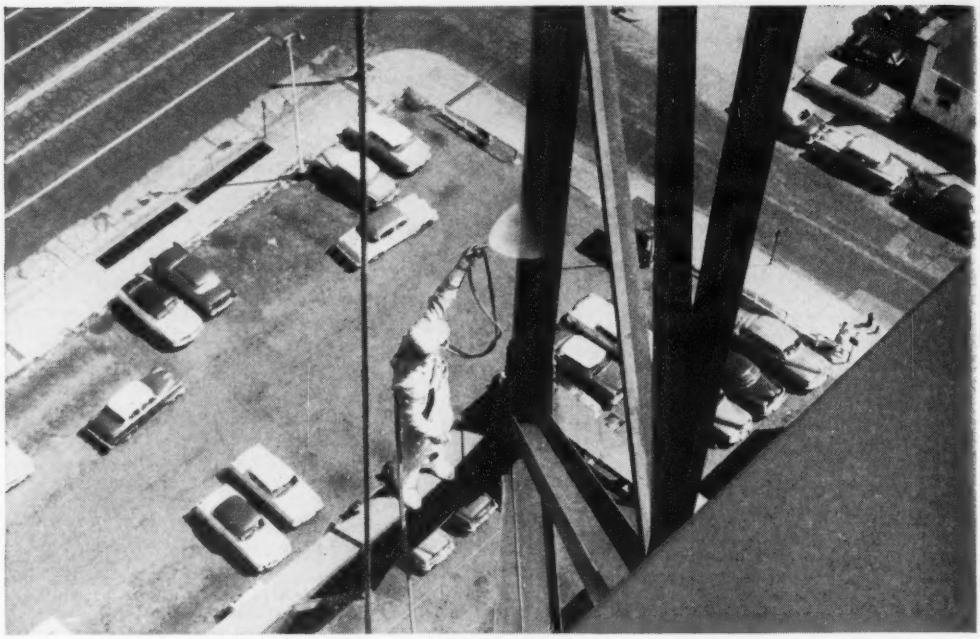
Corrosion Research in Progress by G. A. Marsh and E. Schaschl

TECHNICAL TOPICS SECTION

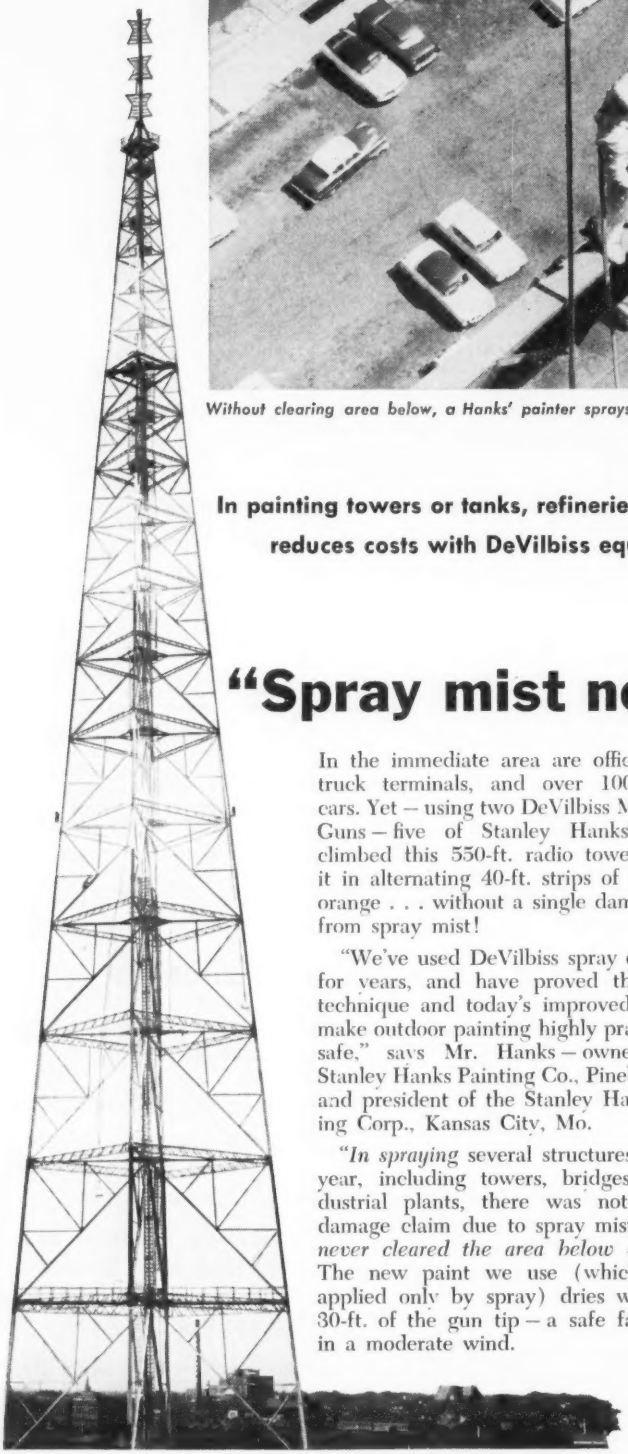
Controlling Corrosion of Textile Mill Air Conditioning Equipment by J. Stanley Livingstone

Chemical Surface Preparation of Structural Steel After Erection by Christopher D. Coppard, John H. Lawrence and Henry W. Adams

Epoxy Coatings Perform Well on Steel Exposed to Chemicals, Corrosive Atmospheres by William M. Brackett



Without clearing area below, a Hanks' painter sprays 550' radio tower.



In painting towers or tanks, refineries or plants—noted contractor reduces costs with DeVilbiss equipment, states . . .

Stanley Hanks

"Spray mist no longer a problem"

In the immediate area are offices, stores, truck terminals, and over 1000 parked cars. Yet – using two DeVilbiss MBC Spray Guns – five of Stanley Hanks' painters climbed this 550-ft. radio tower, painted it in alternating 40-ft. strips of white and orange . . . without a single damage claim from spray mist!

"We've used DeVilbiss spray equipment for years, and have proved that proper technique and today's improved materials make outdoor painting highly practical and safe," says Mr. Hanks – owner of the Stanley Hanks Painting Co., Pinelawn, Mo., and president of the Stanley Hanks Painting Corp., Kansas City, Mo.

"*In spraying* several structures this past year, including towers, bridges, and industrial plants, there was not a single damage claim due to spray mist. *And we never cleared the area below our work!* The new paint we use (which can be applied only by spray) dries well within 30-ft. of the gun tip – a safe factor even in a moderate wind.

"There are many jobs, indoors as well as out, that can be handled only by spray, and others where if given the choice we prefer the gun over other methods. For DeVilbiss spray reduces painting costs; provides smoother, better-covering finishes. And because it's much faster, DeVilbiss spray lets you do a complete job on a weekend or at night, so as not to interrupt a company's work schedule."

How about you? Whether your painting assignments are indoors or out, whether surfaces are rough, smooth, flat or irregular – you'll apply better finishes, in less time, at lower cost, with DeVilbiss spray. Write for *Catalog M-50.* Or call your DeVilbiss supplier. *The DeVilbiss Company, Toledo 1, Ohio. Offices in principal cities.*

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30 Papers Scheduled for Western Region Meeting

Seven Technical Papers, Two Round Tables, Two Plant Tours Scheduled at Birmingham

Seven technical papers, two round table discussion sessions and two plant tours are scheduled events of the October 10-11 Southeast Region meeting at Birmingham, Ala. W. H. Stewart, Sun Pipe Line Co., Beaumont, Texas, NACE president will give the opening address. Registration fee is $5.

The program is:

Thursday, October 10

Liquid Metals Corrosion, Richard Ellis, Southern Research Institute.

Underground Corrosion and Its Mitigation by Use of Coal Tar Coatings, Frank J. Kiernan, Barrett Division, Allied Chemical & Dye Corp., New York.

PVC Pipe, Valves and Fittings, S. A. Lewis, Walworth Corp.

Teflon Resins, E. I. duPont de Nemours & Co., Inc.

Round Table Discussion.

Friday, October 11

Sources of Underground Corrosion Potentals, K. G. Compton, Bell Telephone Laboratories.

Cathodic Protection Systems, Application and Limitations, Wayne Johnson, Corrosion Rectifying Co.

Interesting Corrosion Problems I Have Worked On, Robert J. Kuhn, engineering consultant.

Round Table Discussion.

Tours will be offered between 1:30 and 4:30 pm October 11 to Fairfield Plant, Tennessee Coal, Iron and Railroad Co. or Southern Research Institute.

Other events on the program include showing of a motion picture on corrosion; a business meeting of Southeast Region during the afternoon and the Fellowship Hour and banquet on the evening of October 10.

SECTION CALENDAR

Oct.

1 Shreveport Section. Washington-Youree Hotel.
9 Tidewater Section.
29 Philadelphia Section. Poor Richard Club. Panel discussion: Materials of Construction.
29 Southwestern Ohio Section, Dayton, Ohio.

Nov.

5 Shreveport Section. Washington-Youree Hotel.
19 Chicago Section. Field Evaluation of Pipeline Coatings, M. E. Parker, Consulting Engineer, Houston.
26 Southwestern Ohio Section. Chemical Corrosion, sponsored by National Distilleries. At Cincinnati.

Puget Sound Section Is Organized at Seattle

Puget Sound Section, organized among about 50 NACE members in the vicinity of Seattle, Washington, is the 55th local section in NACE. Mark W. Battersby, Kirk Engineering Co., Seattle, has been named first chairman. Other officers are Robert T. Mercer, R. T. Mercer, Inc., Seattle, vice-chairman and Frank Nisbet, Shell Oil Co., Anacortes, Wash., secretary-treasurer.

Fifth meeting of the group was held September 5 at Seattle.

San Diego Program Also Includes Two Round Table Sessions

Thirty technical papers, two panel discussions on pipe materials and a well-rounded entertainment program have been scheduled for the October 23-25 Western Region Seventh Annual Conference. It will be held at the U. S. Grant Hotel, San Diego.

Registration will begin Wednesday morning. Opening ceremonies include addresses by Charles G. Dail, San Diego mayor; Linn E. Magoffin, Western Region chairman and W. H. Stewart, NACE president. Registration fee is $12, including the Thursday evening banquet. Advance registrations will be received by Kenneth R. Christy, P. O. Box 695, LaMesa, Cal., registration chairman. Hotel reservations should be made directly with the hotel.

The program has been divided into three categories. General interest subjects—all to be presented Wednesday afternoon. Utilities Sessions—Thursday and Friday morning and afternoon concurrently with an alternate program.

A general summary of the information to be presented follows:

GENERAL INTEREST SUBJECTS

Wednesday Afternoon

Role of corrosion engineer in industry, fundamentals, economics and the seabottom as an environment for metal corrosion.

UTILITIES SESSIONS

Thursday Morning

Panels on service pipe and main pipe materials.

Thursday Afternoon

Chemical corrosion in water systems, bacteriological corrosion, cathodic protection design and practice, San Diego sewerage system corrosion problems.

Friday Morning

New developments in bacteriological corrosion, corrosion in connection with Colorado River water, a review of new attitudes toward electrical grounding design, cathodic protection and oxygen inhibition—concrete bridge reinforcing.

Friday Afternoon

Coatings use by Arizona Public Service Company, painting the tuna fleet, cement coatings and linings, corrosion prevention in water meters.

ALTERNATE SESSIONS

Thursday Morning

Radioisotopes in corrosion control, inhibitors for the control of fresh water corrosion, resume of intergranular cor-

(Continued on Page 100)

Combined Technical Conference, Short Course Opens November 12 at Key Biscayne, Florida

A combined technical conference and corrosion control short course under auspices of Miami, Fla. Section NACE will be held November 12-16 at Key Biscayne Hotel, Key Biscayne, Fla. Registrations will be taken all day November 12. The program includes discussions of fundamentals, carbon, magnesium and aluminum anodes, rectifiers, coatings, aluminum pipelines, and nonmetallic piping.

A social hour, banquet and dance are scheduled Friday, November 15.

A tentative program follows:

Wednesday, November 13

Introduction—Corrosion Short Course, J. B. Prime, Jr., Florida Power & Light Co.

Fundamentals of Corrosion, Sam Peabody, Ebasco Services Inc., New York.

Fundamentals of Corrosion, T. P. May, The International Nickel Co., Inc.

Fundamentals of Corrosion, Raymond F. Hadley, Sun Pipe Line Co.

Thursday, November 14

Fundamentals of Corrosion, K. G. Compton, Bell Telephone Laboratories.

Rectifiers and Associated Engineering, Wayne Broyles, Brance-Krachy Co., Inc.

Carbon Anodes, by a representative of National Carbon Co., Union Carbide Corp.

(Continued on Page 100)

K. G. Compton Will Talk At New York November 6

Atmospheric Corrosion: Contributing Factors, Experience and Testing Techniques is the subject of a presentation by K. G. Compton, Bell Telephone Laboratories, Murray Hill, N. J. scheduled for the November 6 meeting of New York Metropolitan Section.

A four-meeting program issued by the section was as follows:

September 11—Common Conventional Coatings for Corrosion prevention of Underground Metal Structures—A Symposium, Frank J. Kiernan, Barrett Division, Allied Chemical & Dye Corp., New York.

October 2—API Tanker Corrosion Research Project—History, Progress and Future Plans, Donald McLean, Assistant Project Coordinator, American Petroleum Institute.

Dec. 11—Special Christmas Meeting: NACE Corporate Memberships, W. H. Stewart, Sun Pipeline Co. Beaumont, Texas.

All meetings are at Chi Am Chateau, Route 22, Westfield, N. J.

Lehigh Valley Section Elects E. A. Anderson

Officers for the Lehigh Valley Section for 1957-58 are as follows: Edmund A. Anderson, New Jersey Zinc Company, chairman; Kenneth R. Cann, Ingersoll-Rand Company, Phillipsburg, New Jersey, vice-chairman; and William H. Rutter, Electro Chemical Eng. & Mfg. Co., Emmaus, Pa., secretary-treasurer.

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Kretschmer Is Named To NACE Board

W. J. Kretschmer, Columbia Gas System Service Corp., Columbus, Ohio has been named to serve the unexpired term of Hugh Hamilton on the National Association of Corrosion Engineer's board of directors. Mr. Hamilton, who represented active membership, had been scheduled to serve into 1959.

Kretschmer

Mr. Kretschmer, senior corrosion engineer for his company, has had more than 16 years experience in corrosion control work on pipelines, cable and other utility property. A registered Ohio professional engineer, he attended the electrical engineering department of Ohio State University. An early member of NACE, Mr. Kretschmer is active on technical committees, is a member of the American Gas Association's corrosion committee and is past chairman of the AGA subcommittee on underground corrosion.

Combined Technical—

(Continued From Page 99)

Aluminum Anodes, J. B. West, Aluminum Company of America.

Magnesium Anodes, George Donahue, Standard Magnesium Co.

Friday, November 15

Coatings, J. W. Pedlow, Quelcor, Inc.

Comparison of Various Types of Organic Coatings, Fred C. Weber, Phelan-Faust Co.

Epoxy Coatings, by a representative of Amercoat Corp.

Aluminum Pipelines, Underground, Above Ground and Under Water, E. T. Wanderer, Aluminum Company of America, Inc.

30 Papers Scheduled—

(Continued From Page 99)

rosion, cathodic protection of oil wells.

Thursday Afternoon

Cathodic protection of naval vessels, corrosion in offshore drilling rigs, deterioration by marine boring organisms.

Friday Morning

High temperature oxidation, surface treatment, corrosion characteristics of aluminum.

Friday Afternoon

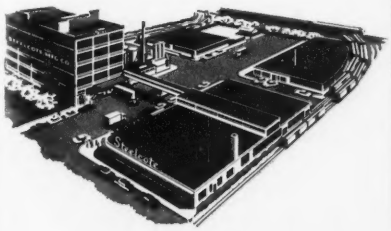
Corrosion in chemical plants, corrosion in a laundry, military ordnance plant corrosion, corrosion of ship yard equipment.

STEELCOTE CORROSION NEWS

1957 Reporting the Latest Developments in Corrosion Control 1957

Steelcote Epoxy Research Has Produced Many New Products

In addition to EPO-LUX 100 and ZINC RICH ZINC DUST PRIMER, Steelcote research has produced epoxy products that create new standards of performance in the fields of floor, grout, and wall repair and resurfacing, tank linings, boat bottom construction and repairs of cracks in metal.



VIEW OF BUILDINGS HOUSING STEELCOTE RESEARCH LABORATORY

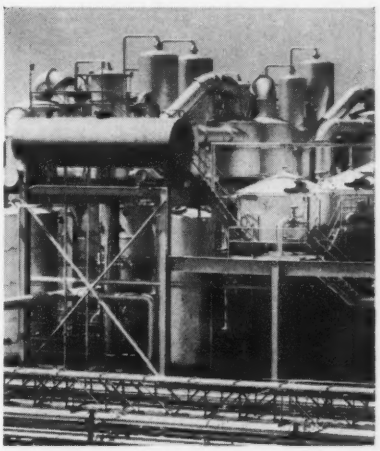
President of Steelcote Invites Corrosion Engineers to Avail Themselves of Steelcote Engineering Service

Steelcote manufactures more than 100 products. For consultation on the use of these products or to solve special problems, we offer you the services of our Engineering Department. Just drop a line describing your problem to Steelcote Manufacturing Co., 3418 Gratiot, St. Louis 3, Mo., U.S.A. In Canada: Steelcote Mfg. Co., Ltd., Rodney, Ontario. Jobbers in principal cities. Write Dept. 6.

Steelcote Epo-Lux Zinc Rich Zinc Dust Primer Licks Industry's Tough Corrosion Problems

Makes Possible Cathodic Protection, Plus Strongest, Longest-lasting Resistance to Acids, Alkalis, Salts and Solvents

Steelcote EPO-LUX ZINC RICH ZINC DUST PRIMER deposits over 90% pure zinc metal to the surface. This amount gives the maximum cathodic action possible from cold "on site" application. Epoxy adhesion and flexibility assures satisfactory performance of an overcoat of EPO-LUX 100. This most resistant of all type air dry organic coatings to alkalis, acids, solvents and salts assures the zinc will continue to give cathodic protection for the maximum number of years. In the most extreme conditions Steelcote epoxies are sometimes needed in combination with other pigments as an in-between coat for greastest possible density and imperviousness. We invite you to undertake an immediate test application.



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HARDNESS	75% hardness of glass with high gloss
FLEXIBILITY	Excellent dimensional stability
RESISTANCE TO SALTS, SOLVENTS, ACIDS AND ALKALIS	Superior to all other air dry organic finishes
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FILM THICKNESS	2 to 3 mils per coat when brushed, sprayed or rolled

STEELCOTE EPO-LUX ZINC RICH ZINC DUST PRIMER	
CORROSION—RUST RESISTANCE	Superior to all other primers
ELECTRO-CHEMICAL ACTION	Protects by cathodic blanketing
FILM COMPOSITION	90% + Pure Zinc metal
ADHESION	Superior to all others because of exclusive epoxy impregnation

NACE aims to use CORROSION as a medium for coordinating corrosion control work the world over.

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- Active and Junior NACE members and Companies seeking employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point text type.
- Advertisements to other specifications will be charged for at $10 a column inch.

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Salary commensurate with ability and experience. Age limit 40.

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First Corrosion Research In Progress Article Ready

First of a series of articles under the heading "Corrosion Research in Progress" is scheduled for publication in November CORROSION's Technical Section.

Preparation and publication of these articles is a function of the National Association of Corrosion Engineers' Committee on Research, of which Aaron Wachter, Shell Development Co., Emeryville, Cal. is chairman. Julius J. Harwood, Head, Metallurgy Group, Office of Naval Research, Washington, is specifically in charge of the preparation and publication of the articles.

The series is intended to stimulate widespread exchange of information about corrosion research underway. Articles are designed to occupy about one page in CORROSION (about 1000 words) and will discuss objectives, methods of attack, preliminary results and other interesting information about research in progress. Mr. Harwood is interested in receiving contributions for the series and will supply more information on request.

American Motors Plans To Dip Its Auto Bodies

Steel magazine reports in its August 28 issue that American Motors Company will dip its 1958 bodies into a tank containing red iron oxide fortified with zinc chromate. This is expected to coat areas inaccessible to spray painting with a corrosion resistant material.

SILICONE ELECTRODE

A new and better way to find Holidays in Dielectric Coating

This new Conductive Silicone Electrode, used in testing coated surfaces, overcomes the problems encountered with wire brush electrodes. It will not "wet" in hot coating material and remains clean at all times. The tough, rubber-like consistency of the silicone makes it wear indefinitely. It will retain its shape and conductivity in temperatures from −20° F to +450° F. Flexible to conform to surface irregularities, this electrode is recommended for use with all types of holiday detectors using high voltage electrical energy. Electrodes are available in widths of 2-in., 4-in., 8 in., 12-in., and 16 in. Wand handles of phenolic plastic are available in 18-in. and 36-in. lengths.

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NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1957

Oct. 10-11—Southeast Region Meeting. Dinkler-Tutwiler Hotel, Birmingham, Ala.

Oct. 23-25—Western Region, Seventh Annual Conference, U. S. Grant Hotel, San Diego, Cal.

Oct. 23-25—7th Biennial Permian Basin Corrosion Tour. Midland-Odessa area, Texas.

Nov. 12-14—Northeast Region Fall meeting, Pittsburgh, Pa., Penn-Sheraton Hotel.

1958

Mar. 17-21—NACE Annual Conference, Civic Auditorium, San Francisco, California.

Oct. 5-8—Northeast Region, Somerset Hotel, Boston, Mass.

Oct. 15-17—North Central Region. Cincinnati, Ohio.

Oct. 20-24—South Central Region. New Orleans, Roosevelt Hotel.

1959

NACE Annual Conference, Sherman Hotel, Chicago, Illinois.

Oct. 12-15—South Central Region Meeting, Denver, Col.

SHORT COURSES 1958

October 22-23—Western Region. Corrosion Control Course (Evening Sessions), Hotel U. S. Grant, San Diego, Cal.

November 12-15—Miami Section Short Course, Key Biscayne, Fla.

December 9-13—Biennial Short Course on Cathodic Protection. University of Illinois and NACE.

Stress Corrosion Is Lehigh Valley Topic November 18

A panel will discuss stress corrosion at the November 18 meeting of Lehigh Valley Section at the second of the section's current series of meetings. A discussion of the Wallenpaupack Penstock by Mr. Van Vliet of Pennsylvania Power and Light Co. was the scheduled topic for the September 15 meeting.

Other meetings have been set as follows, all at Village Inn, Allentown, Pa. except as indicated otherwise:

January 20—Beryllium.

March 17—Corrosion of Iron and Steel.

May—Field trip, Glidden Company, Reading, Pa.

Steinert on Program

W. W. Steinert, A. V. Smith Engineering Co., Narberth, Pa. will be the technical speaker at an October 2 meeting of Southern New England Section at Hartford, Conn.

•

Abstracts in CORROSION come from 28 agencies.

•

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Chemfast is a heavy-duty coating for protecting indoor and outdoor surfaces of wood, metal and masonry against moisture, acids and alkalis. It owes its toughness to Devran (epoxy resin).

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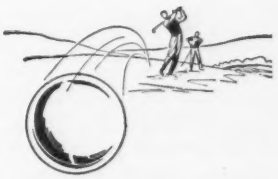

MADE WITH DEVRAN— IT'S TOUGHER, MORE RESILIENT THAN A GOLF BALL

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Pittsburgh Nov. 12-14 Meeting Program Completed

Variety of Topics Included Among Technical Papers

Nineteen technical papers and one demonstration have been arranged for the technical program to be given at the Northeast Region's November meeting. The three-day conference will be held at Pittsburgh's Penn-Sheraton Hotel, with the first day devoted to technical committee meetings. Eight plant tours have been scheduled.

Subject matter of the symposia is varied to match the interests of the region. Symposia are scheduled on underground corrosion, chemical industry, protective coatings, new metallurgical developments, power industry and instruments. A demonstration showing electrochemical reactions is scheduled also.

Subjects to be discussed during meetings of technical committees were listed in August CORROSION on Page 83.

Bobalek

Cogshall

Costanzo

Cox

Erickson

Francis

Halbig

Hess

Husock

Kendall

Manly

McIlrath

McVey

Mears

Noll

Phelps

Renshaw

Sprowls

TECHNICAL PROGRAM

Titles and Abstracts

Wednesday, November 13

Underground Corrosion

A Demonstration of Electrochemical Corrosion Reactions by the Use of Transparent Cells in an Optical Projector, George C. Cox, Consulting Engineer, Charleston, W. Va.

Development of miniature transparent cells resulted from a need to demonstrate the correct and incorrect uses of cathodic protection on coated underground pipe line networks and related structures. These cells have been used to advantage in studying various electrochemical changes or reactions such as ionic migration rates, electrode reactions, electroplating reactions, etc.

Presentation will be essentially confined to an illustration of various corrosion reactions and of methods of preventing or inhibiting such reactions.

Weather vs. Cathodic Protection of Underground Pipeline, Frank E. Costanzo, Manufacturers Light & Heat Co., Pittsburgh.

A discussion of the accumulation of weather data and cathodic protection survey data of a four-mile section of 20-inch bare pipeline in southwestern Pennsylvania over a period of five years. Relationship between rectifier current output and rainfall, soil temperature and mean daily temperatures will be discussed.

Corrosion, Cathodic Protection and Common Sense, Bernard Husock, Harco Corp., Cleveland.

Techniques employed in combatting corrosion are discussed and examples are given to show that some methods can aggravate a corrosion condition. The view is taken that very often "common sense" used in evaluating a corrosion problem can lead to erroneous solutions and that a more complete use of basic corrosion principles is required. It is also shown that by using "common sense" we can fail to recognize corrosion problems. The use and abuse of common sense techniques in cathodic protection are discussed.

Power Industry

Limitations on Chemical Means of Controlling Corrosion in Boilers, Douglas E. Noll, Hall Laboratories Div., Hagan Chemicals and Controls, Inc., Pittsburgh.

Many tube failures in high pressure boilers result from corrosion of the boiler metal or from overheating caused by insulating deposits of corrosion products carried into the boiler. Many factors bearing upon corrosion in power plants are recognized and generally their effects can be overcome by suitable chemical treatment but boiler inspections occasionally reveal unusual magnetic iron oxide deposits whose presence is not always easy to explain in the light of accepted theories of boiler water conditioning. Recent laboratory experiments may provide the answer if we are willing to permit modification of old theories. Further laboratory investigation more nearly approximating actual boiler performance is suggested, but presently there is the implication that the water chemist and boiler plant operator are confronted with a "Hobson's choice" when they consider using chemicals for corrosion control.

Under certain operating conditions periodic acid cleaning of boilers is essential if tube failures are to be avoided.

Fundamentals of Liquid Metal Corrosion, W. D. Manly, Oak Ridge National Laboratory, Oak Ridge, Tenn.

During the past few years interest in liquid metals as coolants and heat transfer systems in

(Continued on Page 105)

TECHNICAL PROGRAM
Titles and Abstracts

(Continued From Page 104)

power production and atomic energy work has been greatly magnified. This has resulted in intensified research to determine compatibility of the various structural metals with these unique coolants.

Corrosion of structural metals in liquid metals is for the most part due to solubility of the various constituents of the metal or alloy in the liquid metal. The manner in which this solution manifests itself gives rise to many types of attack, ranging from a simple solution type attack to a deep intergranular attack with the preferential leaching of one constituent of an alloy. Examples of the many types of corrosion experienced with solid metal-liquid metal systems will be illustrated. Role of impurities on corrosion will also be discussed.

In addition to the solution step, which would soon disappear in a static-one metal system due to reaching a solubility limit, the corrosion of metals can be continued by removal of materials from solution in the liquid metal with a temperature gradient or by dissimilar metal transfer. Thus transfer of metal from hot region of a plumbing system with its deposition in the cold zone and the transfer of material from one part of the system and its alloying with a metal of different composition in another part of the system will greatly increase the corrosion as compared to results obtained in static systems.

Examples of temperature gradient mass transfer and dissimilar metal transfer will be reviewed and factors governing the occurrence of these phenomena will be outlined.

Corrosion of Copper and Copper Alloy Heat Exchanger Tubes in Exhaust Condensers, D. S. Hibbard, American Brass Co., Buffalo, N. Y.

Copper and its alloys have been used widely in central power stations. Various environments may affect the service of copper and its alloys. Types of corrosion most likely to affect copper alloys are dezincification, stress corrosion cracking, pitting corrosion, corrosion fatigue, impingment corrosion and galvanic corrosion. To meet requirements of certain conditions, alloys with certain properties have been developed.

Because many factors may combine to cause corrosion of condenser tubes, as much accurate information about operating conditions as possible should be available for proper analysis of the problem.

Instruments

Development of the Redox Probe—Field Technique, Frank E. Costanzo and Roger E. McVey, Manufacturers Light and Heat Co., Pittsburgh.

Further information is given on the Redox Probe developed by A.G.A. Presentation of a new rugged probe and the procedure for tests will be made. Redox data will be compared to: 1. Pipe-to-$CuSO_4$ profile study. 2. 4-point soil resistance survey. 3. pH readings. 4. Earth current readings and 5. Seasonal variation. Another survey will compare Redox readings to pit depth.

Electrochemical Mapping of Corrosion Currents, Howard T. Francis, Armour Research Foundation, Chicago.

A technique is described for plotting the anode-cathode distribution over the surface of a corroding specimen. A cylindrical specimen is rotated at 1725 rpm in the corrosion medium. A small reference electrode placed near the surface of the cylinder senses the potential differences in the electrolyte induced by the corrosion currents. The scanning electrode is simultaneously moved parallel to the axis of rotation, so that an area of the cylinder is scanned. The potential differences are amplified and presented on the Z axis of an oscilloscope, while vertical and horizontal distances on the specimen are presented on the X and Y axes. Thus, a picture is produced which closely resembles a television picture, white and black regions representing anodic and cathodic zones of the specimen, respectively.

Coating Evaluation, Harold C. O'Brien, Royston Laboratories, Blawnox, Pa.

Abstract not available.

Chemical Industry

Design Related to the Application of Protective Coatings, K. G. LeFevre, Metalweld, Inc., Philadelphia.

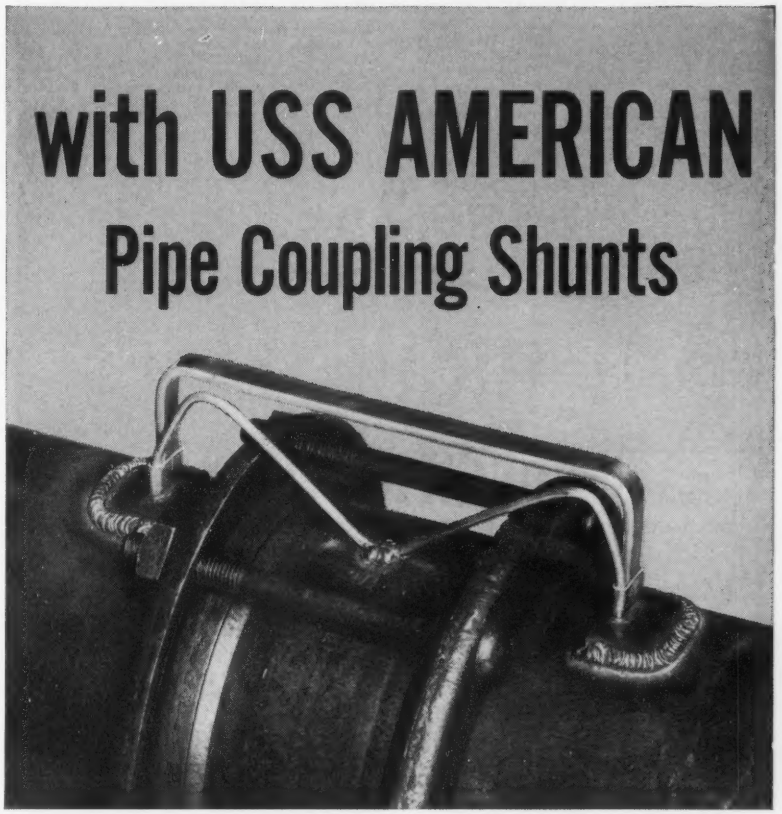
Abstract not available.

Protective Coating Standardization in a Multi-Plant Chemical Operation, S. W. McIlrath, Diamond Alkali Co., Cleveland.

Abstract not available.

(Continued on Page 106)

Fight corrosion and save money



They're prefabricated, and curved to fit the pipe! USS American Pipe Coupling Shunts are designed especially for easy application on mechanical pipe couplings. They save the time and labor of fabricating home-made shunts, are easier to install, and do a better job.

Terminals and secondary conductors are steel. Main conductor is heavy copper bar. Conductors are welded permanently to the pipe and coupling to keep entire assembly at same potential.

Write for information about cathodic protection devices manufactured by American Steel & Wire. These include pipe coupling shunts; anode connectors; suspended anode connectors for piers, dry docks, ships; and coupon connectors for connecting leads to pipe.

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GENERAL OFFICES: CLEVELAND, OHIO
COLUMBIA-GENEVA STEEL DIVISION, SAN FRANCISCO, PACIFIC COAST DISTRIBUTORS
TENNESSEE COAL & IRON DIVISION, FAIRFIELD, ALA., SOUTHERN DISTRIBUTORS
UNITED STATES STEEL EXPORT COMPANY, NEW YORK

USS

USS AMERICAN PIPE COUPLING SHUNTS

UNITED STATES STEEL

TECHNICAL PROGRAM
Titles and Abstracts

(Continued From Page 105)

The Use of Electrochemical Measurements to Predict Corrosion Rates, R. B. Mears, U. S. Steel Applied Research Laboratories, Monroeville, Pa.

Various types of electrochemical measurements used or suggested for use in predicting corrosion rates will be described and evaluated. These range from direct measurement of potential of local corrosion cells and of the quantity of corrosion current to indirect measurements based on fundamentally or empirical established relationships.

It will be pointed out that while many of the procedures have shown a good correlation between electrochemical measurements and corrosion rates under selected laboratory conditions, none of the methods are generally applicable for field use at present. Furthermore, it will be pointed out that even the best of these types of measurements yield misleading indications in special cases. As a result of recent advances in electronic techniques and in the knowledge of mechanism of corrosion, it is believed that a sustained attack on the instrumentation problem would yield successful results.

Evaluation of Corrosion Data by Mathematical Statistics, V. V. Kendall, National Tube Division, U. S. Steel Corp., Pittsburgh.

Abstract not available.

Protective Coatings

Surface Preparation Techniques, J. H. Cogshall, Pennsalt Chemicals Corp., Philadelphia.
Abstract not available.

A Modern Approach to Corrosion Prevention Through Protective Coatings, H. W. Howard, Shell Chemical Co., Cleveland.
Abstract not available.

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The Search for Steam Resistant Coatings, E. G. Bobalek, W. R. R. Park and E. G. Bell, Case Institute of Technology, Cleveland.

Steam exposure presents the most severe environment for producing blistering failure of synthetic resin coatings. Conflicting evidence has been presented over the past several years regarding the performance of modern coatings in preventing the corrosion of steel in steam exposure. An experimental survey was made by panel testing more than 120 commercial coatings representing about 25 different chemical types over a variety of surface treatments. Tabular comparisons are made regarding the relative worth of different types of coatings. A number of coating types, properly applied, can withstand 500 hours or more of exposure to 250 F steam.

Some theoretical conclusions are advanced which suggest how important variables of coating formulations can effect steam and blistering resistant properties.

Corrosion Protection of Power Plants by Organic Coatings, P. M. Hess, Safe Harbor Water Power Corp.

An account of a development program, with a few illustrations, carried out over a period of years on a practical basis to improve the very unsatisfactory service received from protective coatings available at the time. Considerable emphasis is placed on improvements in organic coatings during the period of research and progress in verifying the need for proper preparation of the surfaces prior to applying the coatings, especially when used to protect ferrous underwater equipment. Value of methods of application is enumerated in relation to the cost and service life of the product used, together with the cost of each operation required for the overall job related to a per year life basis.

The author points out some difficulties experienced with steel fabricators in having them apply specified other than their standard shop coats.

Use of color schemes to enhance plant architecturally, improve safety, better employee morale and improve operating efficiency are enumerated as well as the desirability of utilizing the heat conductivity, and heat and light reflecting qualities of a product whenever its durability as a protective coating is satisfactory.

Final evaluation points out the need for determining the simplest and cheapest procedure and selecting a material which will do the best protection job at a particular location with a compromise on the other desirable requirements associated with protective and beautifying coatings.

Thursday, November 14

New Metallurgical Developments

Some Newer Developments in Corrosion Resisting Steels and Steel Products, John Halbig, Armco Steel Corp., Middleton, Ohio.

This paper describes a number of new steels and steel products. The design of each is based at least in part on a requirement for corrosion resistance. These products embrace a wide field of interest and are the result of active industrial research.

Several precipitation hardening stainless steels give strength and fabricating properties not heretofore available in corrosion resisting materials. While certain of these steels have wide application in the aircraft industry, they are also used for many other purposes. This diversity of application is discussed. Data are also presented on a new austenitic stainless steel being widely used in automotive valves.

A new type of aluminum-coated steel is described and compared with its earlier counterpart. The new Type 2 coating finds wide use in atmospheric exposure. The earlier Type 1 coating is used principally where heat is encountered.

Porcelain enamel coatings are now being applied to aluminized steel for decorative purposes, the result being a doubly protected product with excellent strength characteristics. The story of this development is briefly outlined and results are shown.

In the non-metallic coatings field a new corrugated pipe product is finding wide acceptance in municipal and industrial sewage disposal systems. This unique pipe is shown and discussed.

Effect of Metallurgical Factors on Resistance to Corrosion of Aluminum Alloys, D. O. Sprowls, Alcoa Research Laboratories, New Kensington, Pa.

Abstract not available.

(Continued on Page 107)

TECHNICAL PROGRAM

Titles and Abstracts

(Continued From Page 106)

Properties of AM350, William Renshaw, Allegheny Ludlum Steel Co., Brackenridge, Pa. Among the newer materials of construction, AM-350 offers a unique combination of properties. It possesses high strength which can be developed by freezing, a departure from the conventional high temperatures employed with commercial stainless steels. Because of its alloy content, AM-350 has good corrosion resistance, exceeding that of the hardenable 400 series of stainless. This paper discusses resistance to general corrosion as well as localized attack. Mechanical properties are briefly covered for conditions in which corrosion data are reported.

BIOGRAPHIES

Pittsburgh Meeting Authors

EDWARD G. BOBALEK—Professor of chemical engineering, Case Institute of Technology, in charge of coatings and plastics research laboratories. He specializes in theoretical problems on engineering application of polymeric materials. He has a BS from St. Mary's College, 1939; MS Creighton University, 1940 and PhD Indiana University, 1942.

CARL A. ERICKSON, JR.—Since 1947 corrosion engineer for the People's Natural Gas Co. of Pittsburgh, he has an AB from Hobart College, 1947 and an MS, University of Chicago, 1943. Presently secretary of the Western Pennsylvania Corrosion Committee, he is active in NACE technical committee work presently being chairman of T-7 Corrosion Coordinating Committee and of T-7A Northeast Region Coordinating Committee among other activities.

T. G. BELL—Currently project leader in charge of the paint testing program at Case Institute of Technology, he joined the institute after several years with Canadian Industries, Ltd. where he specialized in formulation and testing of synthetic resins.

JAMES H. COGSHALL—Since 1950 with Pennsalt Chemicals Corp., Philadelphia he has been occupied principally with coatings problems. He has been with the company's corrosion engineering products department in Philadelphia, Pa. since August, 1953. He has a BS in chemical engineering, University of Pennsylvania, 1938.

FRANK E. COSTANZO—Senior corrosion engineer with the Pittsburgh Group of Columbia Gas System. He has been working on underground corrosion problems since 1945. Formerly with the Utility Survey Commission from 1936 to 1940, he has a BS in mechanical engineering from University of Pittsburgh, 1933. Long active in NACE affairs, he presently is director representing Northeast Region on the NACE board of directors.

GEORGE C. COX—A consulting engineer on corrosion prevention methods and processes, he has a BS and MS in electrical engineering from North Carolina State College, 1917 and 1926; did graduate work on metallurgy at Columbia University in Applied electronics and electrochemistry at Sorbonne University, Paris. He also did graduate work on industrial and manpower mobilization at the Army Command and General Staff School and the Army War College, followed by a two year assignment to the War Department General Staff on World War II operational planning. While in charge of a corrosion prevention project in the European Theater of Operations he developed an electrolytic method of descaling tetraethyl lead-containing rust layers from gasoline carrying tankships which made possible transportation of lead-free potable drinking water to U. S. troops at a time when retreating Germans were contaminating water supplies. He also helped develop the electrical method of producing a calcareous coating on steel submerged in sea water. He is a retired colonel of the Army of the United States.

HOWARD T. FRANCIS—Supervisor of the Electrochemistry Section of the Armour Research Foundation, he received a BS in chemistry from Mount Union College in 1938 and a PhD from Pennsylvania State College in 1942 when he joined the foundation. His fields of interest include corrosion, electrodeposition and batteries with major emphasis on new solutions of electrochemical problems.

(Continued on Page 108)

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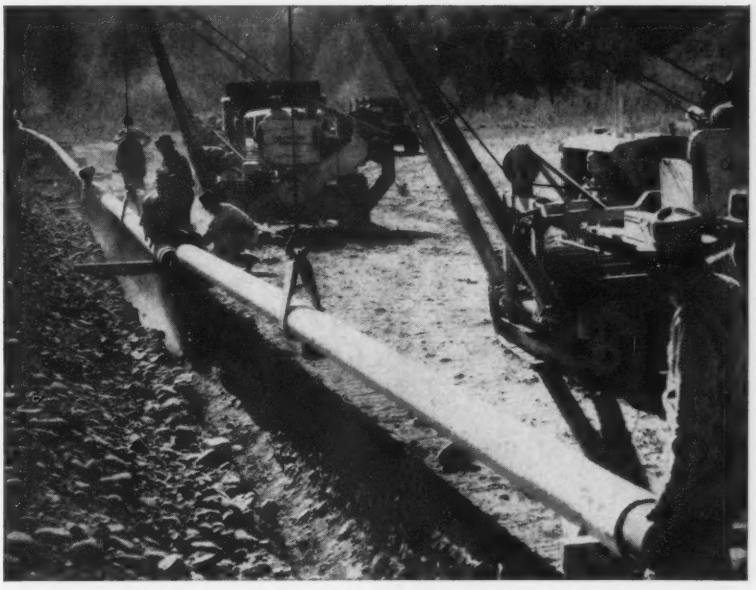

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BIOGRAPHIES
Pittsburgh Meeting Authors

(Continued From Page 107)

JOHN HALBIG—Now senior research engineer with his company, he has been active in corrosion research since 1940 except during a tour of duty with the Corps of Engineers in World War II. He is engaged in industrial corrosion control work, including materials selection and maintenance. He is active in NACE technical committees and otherwise. He has a BS in chemical engineering from Johns Hopkins University, Baltimore and is a graduate of the Army Command and General Staff School, Ft. Leavenworth, Kansas, 1945. He is active also in ASTM and AISI.

P. M. HESS—Presently manager of the Safe Harbor Water Power Corp., he is past chairman of the Engineering Section, Pennsylvania Electric Association and past chairman of the Hydraulic Power Committee of the Edison Electric Institute. He has AB and EE degrees from Swarthmore College. He has been employed by General Electric Company and in 1922 joined Pennsylvania Water and Power Company as a test engineer, later becoming operating engineer. In 1931 he joined his present employers as superintendent of its Safe Harbor Plant, where he combated pitting in Kaplan turbines by using stainless steel and studied means of better coatings protection for underwater structures.

D. S. HIBBARD—Since 1954 metallurgical engineer, Buffalo Division, American Brass Company, principally working on applications of copper alloy heat exchanger tubes and copper alloys in the electrical equipment field. He joined American Brass' Buffalo Division in 1941 where he worked in the technical department until 1946 and from 1946 to 1954 as sales representative for the Buffalo office. He is a graduate of Wesleyan University, Middletown, Conn. and is a member of NACE, AIME and ASM.

BERNARD HUSOCK—Chief engineer since 1953 of Harco Corporation's corrosion control activities, he is a member of AIEE, NACE, Tau Beta Pi and Eta Kappa Nu. Before joining Harco in 1950 he did design and development work on aircraft radar equipment for Philco Corp. He has a BS in electrical engineering from Drexel Institute of Technology and has done graduate work in electronics at University of Pennsylvania.

V. V. KENDALL—For more than 30 years corrosion engineer for National Tube Division, U. S. Steel Corp. he is active in NACE, AISI, ASTM and is a member of other technical organizations. He has held numerous positions ranging from chemist for a railway company to college professor. He has an AB from Missouri Wesleyan College, BS in Ed, University of Missouri and MA in chemistry, University of Missouri.

W. D. MANLY—Associate director of the Metallurgy Division of Oak Ridge National Laboratory engaged in problems associated with atomic reactor development. He is a member of NACE, AIMME, American Nuclear Society and ASM. He has BS and MS degrees from University of Notre Dame. He was with the Marine Corps during World War II in the South Pacific and in China and has held numerous offices in the societies to which he belongs and is author of several papers in his field.

S. W. McILRATH—Central Engineering Dept., Diamond Alkali Co., Cleveland. He has been corrosion specialist for the last three years for all Diamond plants as well as chairman of the company's alkali protective coating standardization program. His corrosion experience includes three years as liaison section on packaging tanks and parts for overseas shipment in U. S. Army's Cleveland Ordnance District and seven years as corrosion engineer at the Painesville, Ohio works of Diamond Alkali. He has a BS in ChE from Fenn College, Cleveland and during 1944-46 was in the Navy Bureau of Ordnance as cognizance officer for rocket propulsion.

R. E. McVEY—He has worked on underground corrosion problems since 1955 as a member of the engineering department of the Pittsburgh Group, Columbia Gas System. He has a BS in biological science, Marshall College, Huntington, W. Va., 1951, is a lieutenant in the naval reserve and a member of NACE.

DOUGLAS E. NOLL—Currently staff engineer for Hall Laboratories' Division, Hagan Chemicals and Controls, Inc., he has been with his employer 14 years. He has had extensive practical experience with water corrosion problems, has worked in the company's research laboratories and directed a quality control program for production of water treating chemicals. He has a chemical engineering degree from Carnegie Institute of Technology and is a registered professional engineer in Pennsylvania.

W. R. R. PARK—Presently with Dow Chemical Company, Midland, Mich., he was in charge of experimental work at Case Institute of Technology between July, 1955 and February, 1957. He is a graduate of University of Toronto and came to Case as a research associate after two years as research chemist with Canadian Industries, Ltd.

E. H. PHELPS—Head of the Corrosion Kinetics Section, Applied Research Laboratory, U. S. Steel Corporation, Monroeville, Pa. He received a BS from Clarkson College of Technology, Potsdam, New York and a MS and

(Continued on Page 110)

TECHNICAL COMMITTEE MEETINGS
NOVEMBER 12, 1957
NORTHEAST REGION MEETING
Nov. 12-14, 1957
Penn-Sheraton Hotel, Pittsburgh

Morning

- T-2E Internal Corrosion of Products Pipe Lines and Tanks
- T-2K Prefabricated Plastic Film for Pipeline Coatings
- T-4B Corrosion of Cable Sheaths
- T-5A-5 Corrosion by Nitric Acid
- T-5D Plastic Materials of Construction
- T-6F Protective Interior Linings Application and Methods

Afternoon

- T-3C Cost of Corrosion
- T-3H Tanker Corrosion
- T-5A-6 HF Corrosion
- T-6D Industrial Maintenance Painting
- T-7A Northeast Region Corrosion Coordinating Committee.

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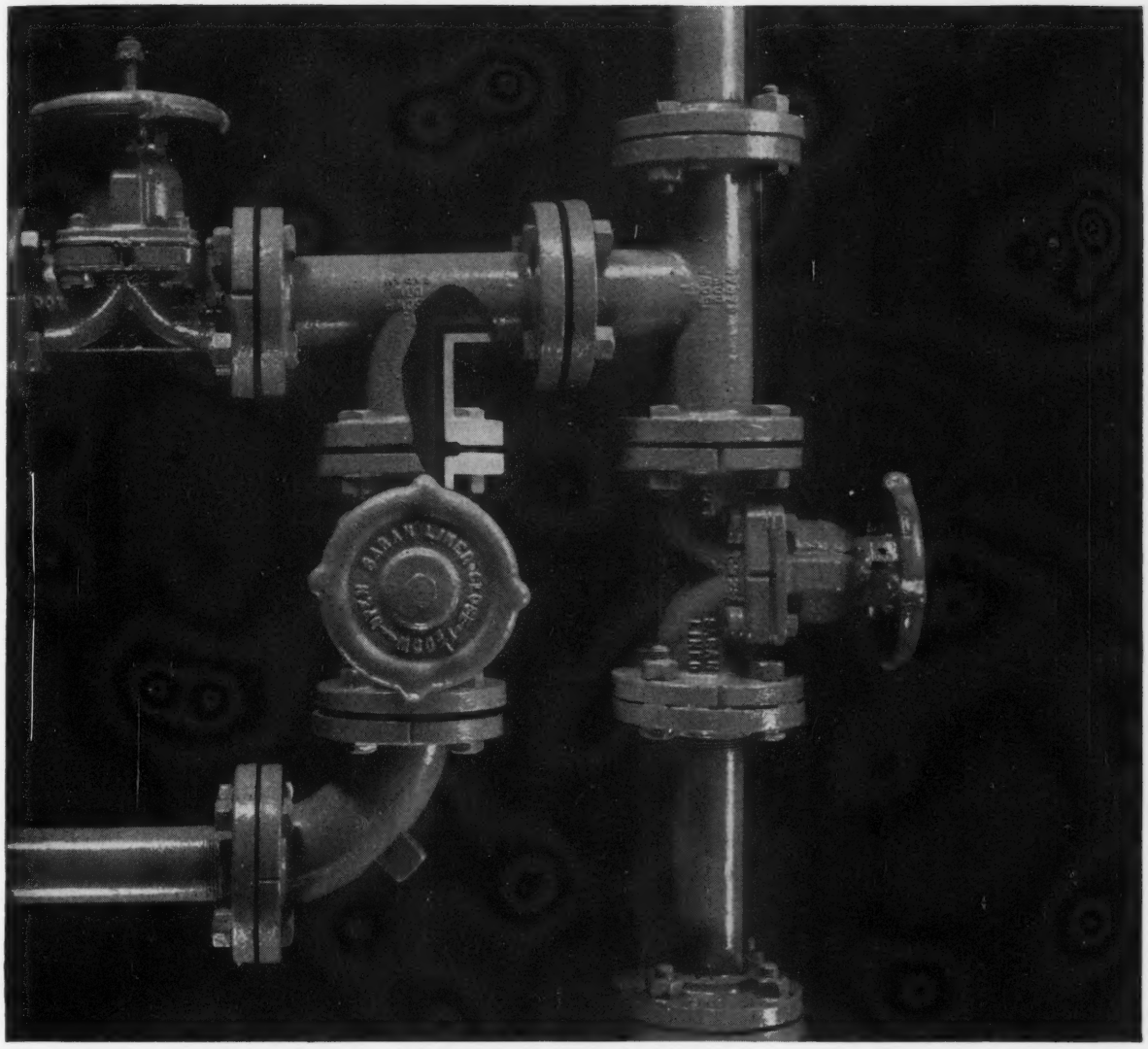

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BIOGRAPHIES
Pittsburgh Meeting Authors

(Continued From Page 108)

PhD from Case Institute of Technology. His experience includes three years in corrosion work at the Materials Laboratory, Wright Air Development Center, Dayton, Ohio. Since joining U. S. Steel, he has been engaged in corrosion research programs on stainless, alloy, and carbon steels.

WILLIAM G. RENSHAW—Supervisory chemical Engineer, Corrosion Section, Allegheny Ludlum Steel Corp. He started work for his present employer in 1940, then spent two years in the Navy working in the electronics field. He is author of several papers on corrosion resistance of stainless steel and titanium.

DONALD O. SPROWLS—He has spent eighteen years in corrosion control work on aluminum and aluminum alloys, especially in atmospheric exposures at Aluminum Research Laboratories, New Kensington, Pa. He has a BS in chemical engineering, Drexel Institute and has attended Carnegie Institute.

Northeast Region Offers Eight Industrial Tours

Preregistration will be required to take any of the following tours offered Wednesday, November 13 during the Northeast Region meeting at Pittsburgh:

Gulf Research and Development Company, Harmarville. The prospecting, production and uses of oil are shown. Entomology laboratory for study of insecticides is especially fascinating.

U. S. Steel Research Center, Monroeville. Fundamental and applied research laboratories.

Pittsburgh Plate Glass Company, Creighton. Manufacturing of plate glass products from melting to finishing on continuous line.

Continental Can Company, West Mifflin Borough. Modern plant for the manufacture of containers.

H. J. Heinz Company, Pittsburgh. Home of the famous 57 varieties. Food processing and packing.

Aluminum Company of America, New Kensington.

Pittsburgh Coke and Chemical Company, Neville Island. Modern coke plant and chemical recovery operations. Chemicals from coal tar.

Fisher Scientific, Pittsburgh. Development and production of laboratory instruments and equipment. World's outstanding collections of paintings of alchemy.

Air, Sea and Land Corrosion Problems Are Aired at Chicago Transportation Symposium

Nantz

Stevens

Corrosion problems of aircraft, oil tankers and Diesel engines are to be covered during the Transportation Symposium presented at Chicago during the October 2-4 North Central Region meeting there at Hotel Sherman. A fourth paper in the symposium pertains to conductivity tests on zinc dust pigmented coatings.

The Transportation Symposium, with S. K. Coburn, Association of American Railroads as chairman, is one of eight scheduled for the Chicago meeting. Details concerning other papers to be presented were published in CORROSION's September issue beginning on Page 95.

The following papers will be presented during the Transportation Symposium:

Aircraft Corrosion Problems by First Lt. David S. Nantz, Project Engineer, Materials Laboratory, Wright Air Development Center, Dayton, Ohio

Because weight is kept to a minimum, margins of safety in aircraft have been reduced to the point that loss of metal by corrosion is serious. Most corrosion encountered by the Air Force results from salt fog, engine exhaust gas, trapped water, dirt or condensation. Means of combating corrosion on jet engines, structural parts and on new high strength aluminum alloys are discussed.

Laboratory Testing of Railroad Diesel Cooling System Corrosion Inhibitors by J. I. Bregman and D. B. Boies, National Aluminate Corp., Chicago.

Development of test methods for laboratory evaluation of corrosion inhibitors in railroad Diesel engines are discussed with relation to the diversity of metals and wide range of operating conditions encountered.

Flask type tests for screening and development and a recirculating test involving a heat transfer surface designed to more nearly duplicate service conditions and finally evaluate a product were developed.

Results are given for several commercial and experimental inhibitors of the boron-nitrite soluble oil and other types. Results are presented also from tests involving coupling or insulation of the several metals, inhibitor dosage level, water composition, presence of anti-freeze compounds as well as effect of inhibitors on nonmetallic components of system.

Importance of inhibitor behavior under test conditions is discussed in relation to conditions of actual use, indicating good correlation between laboratory and field tests.

Corrosion in Tankers by F. M. Watkins, Sinclair Research Laboratories, Harvey, Ill.

The origin and operation of the American Petroleum Institute's investigation of corrosion in ocean-going tank ships is related. While considerable information has been accumulated which is indicative or qualitative, more quantitative information now is needed.

Cooperative shipboard tests have been planned and data are now being collected on several methods of control.

Conductivity Testing of Zinc Dust Pigmented Coatings by H. L. Stevens, Chemist, Materials Laboratory, Wright Air Development Center, Dayton, Ohio.

The development of a satisfactory conductivity test which can be used as the scientific basis for formulation of zinc rich coatings providing good galvanic protection to steel is described. The test described will determine optimum zinc concentration, film thickness and drying time for any given vehicle.

SPECIAL DELIVERY for liquids and gases in all branches of industry is assured with Kraloy Rigid Polyvinyl Chloride (normal and high impact) Plastic Pipe... with virtually no maintenance or policing. You install Kraloy PVC—and forget it. That's why millions of feet of Kraloy Plastic Pipe are in use today, because Kraloy PVC is inert and can never rust, rot, or corrode, is not subject to electrolytic action, handles abrasives, slurry and most Ph factors, is scale resistant. Superior flow characteristics (C factor = 150+) permit use of smaller diameters. Installation costs 50% less, due to light weight and ease of handling.

Write for complete information and literature.

KRALOY RIGID PVC PLASTIC PIPE NORMAL AND HIGH IMPACT

Kraloy Plastic Pipe Co., 4720 E. Washington Blvd., Los Angeles, Dept. C-107

Subsidiary of the Seamless Rubber Co., a Rexall Drug Co. *Subsidiary*

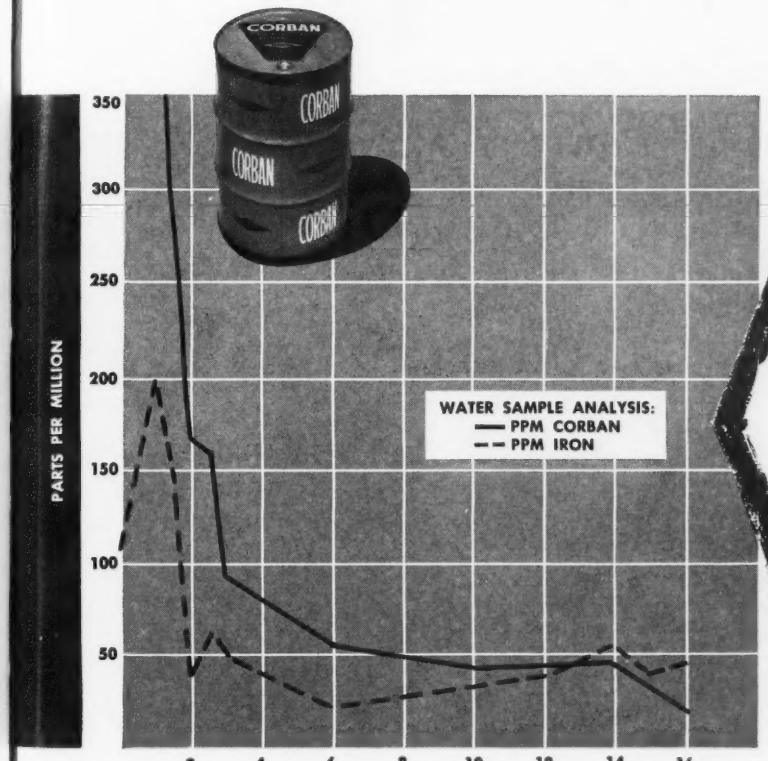
Philadelphia Section to Hold Panel Discussion

A panel discussion on materials of construction will be the featured technical program at an October 29 meeting of Philadelphia Section. Panel members and their specialties are: R. R. Pierce, ceramics, refractories; W. B. DeLong, metallic; Kenneth Chamberlain, structural plastics.

New-and proved in the field

DOWNHOLE CORROSION PROTECTION BY SQUEEZING with CORBAN

Corban® has been recognized for some time as one of the most effective, economical corrosion inhibitors for protecting the internal surfaces of well equipment from corrosion.
Now, research and field testing bring you a new technique for longer-lasting protection. This technique of *squeezing* with Corban involves pumping Corban into the producing formation. It is then slowly produced back with normal production over several months, plating out in a protective film over metal well equipment.
To date this new approach to the use of Corban for corrosion control has been even more successful than anticipated.



Case History of new Squeeze Technique with Corban

Production from this particular gas condensate well is about 10 MMCF per day, with about 150 bbls. of distillate and about one barrel of water. The squeeze treatment with Corban was performed by pumping 50 gallons of Corban 101, mixed with 30 gallons of fresh water, into the formation. This mixture was displaced with condensate and then over-flushed with 12 bbls. of condensate.

All of the pumping was done at a rate of about one barrel per minute. The maximum surface pressure was 5000 psi as the Corban was entering the formation. The pressure while the condensate was pumped into the formation was 4000 psi. No adverse effects on production were noted following the treatment. Corrosion protection was provided for 16 weeks—without having to open up the well for subsequent inhibitor treatments during this period.

Let us recommend a program of well treatment designed especially for your wells. Call any of the 165 Dowell offices in the United States and Canada. In Venezuela, contact United Oilwell Service. Or write Dowell Incorporated, Tulsa 1, Oklahoma.

Services for the oil industry

DOWELL

A SERVICE SUBSIDIARY OF THE DOW CHEMICAL COMPANY

Plastics Symposium for March Meeting Filled

Papers on Pipes and Tanks Scheduled for San Francisco Meet

Extensive information on the physical properties and performance characteristics of principal plastic materials of construction will be divulged during the Plastics Symposium at San Francisco. The symposium, one of 17 to be held during the 14th Annual NACE Conference March 17-21, consists of four papers. It is scheduled to be held on the afternoon of Thursday, March 20.

General content of papers and names of authors in the symposium have been given as follows by W. B. Meyer, St. Louis Metallizing Co. and O. H. Fenner, Monsanto Chemical Co., respectively chairman and co-chairman of the symposium:

A discussion of the fabrication of rigid polyvinyl chloride by Louis Perrett, Jos. T. Ryerson & Sons.

Summary of three years' creep studies on polyvinyl chloride pipe and fittings made in their laboratory by Gordon H. Fornald, Technical Manager and H. H. George, Research Manager of Tube Turns Plastics.

Information on reinforced epoxy pipe and fittings, H. H. Boggs, Fibercast Company, Sand Springs, Okla.

Fabrication of reinforced tanks and ducts by W. E. Smith, Ceilcote Co.

March Exhibition Booth Sales Good

Orders for booth space at the 14th Annual NACE Exhibition at Civic Auditorium, San Francisco, Cal. are being received at Central Office NACE in good volume. This year's descriptive brochure and contract were mailed to an expanded list of prospects somewhat earlier than in previous years.

Up to September 4, contracts for booth space have been received from 17 companies. Additional contracts are being received daily and the number of exhibitors is expected to reach substantial size before the end of the year.

Companies that have contracted for space so far are:

Amercoat Corporation, South Gate, California
Chicago Metallizing Corporation, Chicago, Illinois
Clementina Ltd., San Francisco, California
M. J. Crose Manufacturing Company, Inc., Tulsa, Oklahoma
The Dampney Company, Hyde Park, Boston, Massachusetts
The Duriron Company, Inc., Dayton, Ohio
Gulf States Asphalt Company, Inc., Houston, Texas
The International Nickel Company, Inc., New York, New York
Johns-Manville Sales Corporation, New York City, New York
Lester Equipment Mfg. Company, Inc., Los Angeles, California
F. H. Maloney Company, Houston, Texas
The Pfaudler Company, Rochester, New York
Plicoflex, Inc., Los Angeles, California
Protecto Wrap Company, Tulsa, Oklahoma
U. S. Stoneware Company, Akron, Ohio
Visco Products Company, Houston, Texas
T. D. Williamson, Inc., Tulsa, Oklahoma

More Appointments Made For March Conference

Simmons

Treseder

Personal contacts with individuals and concerns in Western Region who should know about the March 1958 NACE 14th Annual Conference and Exhibition will be directed by William P. Simmons, Alloy Steel Products Co., Inc. Mr. Simmons, assistant to George A. Works, exhibits committee chairman, expects to reach men who otherwise might not learn about the meeting in time to attend.

R. S. Treseder, Shell Development Co., will work with L. E. Habben on the Housing Committee and handle arrangements for the luncheon for NACE corporate members.

P. J. Raifsnider, Shell Development Co., has accepted responsibility for registration. He is a 1941 graduate of University of Wisconsin with a BS in chemistry.

Raifsnider

Plans have been made also for distribution among interested companies over the United States of a poster calling attention to the March meeting.

Lectures Will Be Given on Atmospheric Corrosion

Three educational lectures on atmospheric corrosion, in effect a symposium on the subject, are a planned feature of the NACE 14th Annual Conference at San Francisco. Frederick W. Fink, Battelle Memorial Institute, Columbus, Ohio, chairman of the educational program has scheduled the one-hour lectures on Tuesday, Wednesday and Thursday, March 18-20.

Subject matter will include mechanisms for ferrous and non-ferrous metals, principles of design in atmospheric corrosion experiments, procedures for interpretation of results of atmospheric corrosion experiments, procedures for interpretation of results of atmospheric corrosion experiments.

•

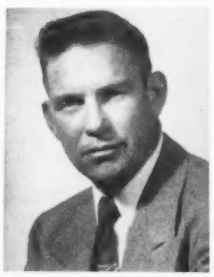
Corrosion publishes annually in December an alphabetical subject index to its Technical Section.

•

A list of available reprint copies of technical reports and articles from CORROSION is available on request.

Announcing Three Complete Locations to Serve You!

HOUSTON. Wayne A. Johnson, President, directs the company's activities from the Main Office in Houston. Mr. Johnson is well known in the cathodic protection profession and is the author of many technical articles in the field. P. O. Box 19177, Houston, Tex.



GREAT BEND, KANSAS. Harvey Fanshier, Mid-Western representative for Corrosion Rectifying Company is a graduate of Kansas State College with extensive experience in salt water disposal and corrosion prevention. Rt. 2, Great Bend, Kansas.

NEW ORLEANS, LA. James T. Condry, New Orleans representative, is a graduate Electrical Engineer in charge of evaluation, design, installation and materials sales for the Louisiana territory. He has wide experience in marine and offshore installations. 2322 Airline Highway, Kenner, Louisiana.

CORROSION RECTIFYING CO., INC.
5310 Ashbrook — Phone MO 7-6659
P. O. Box 19177 — Houston, Texas

An Invitation

GEORGE CHRISTOPHER

OFFICE OF THE MAYOR
SAN FRANCISCO

August 7, 1957

To the Distinguished Members of the
National Association of Corrosion Engineers

Gentlemen:

I have been happy to learn that your important organization is to convene in San Francisco next March 17-21, and I should like to extend to you in advance a special word of invitation and greeting. The work of your organization is most useful and timely, and San Francisco is very glad to be host city for your next annual meeting and exhibit.

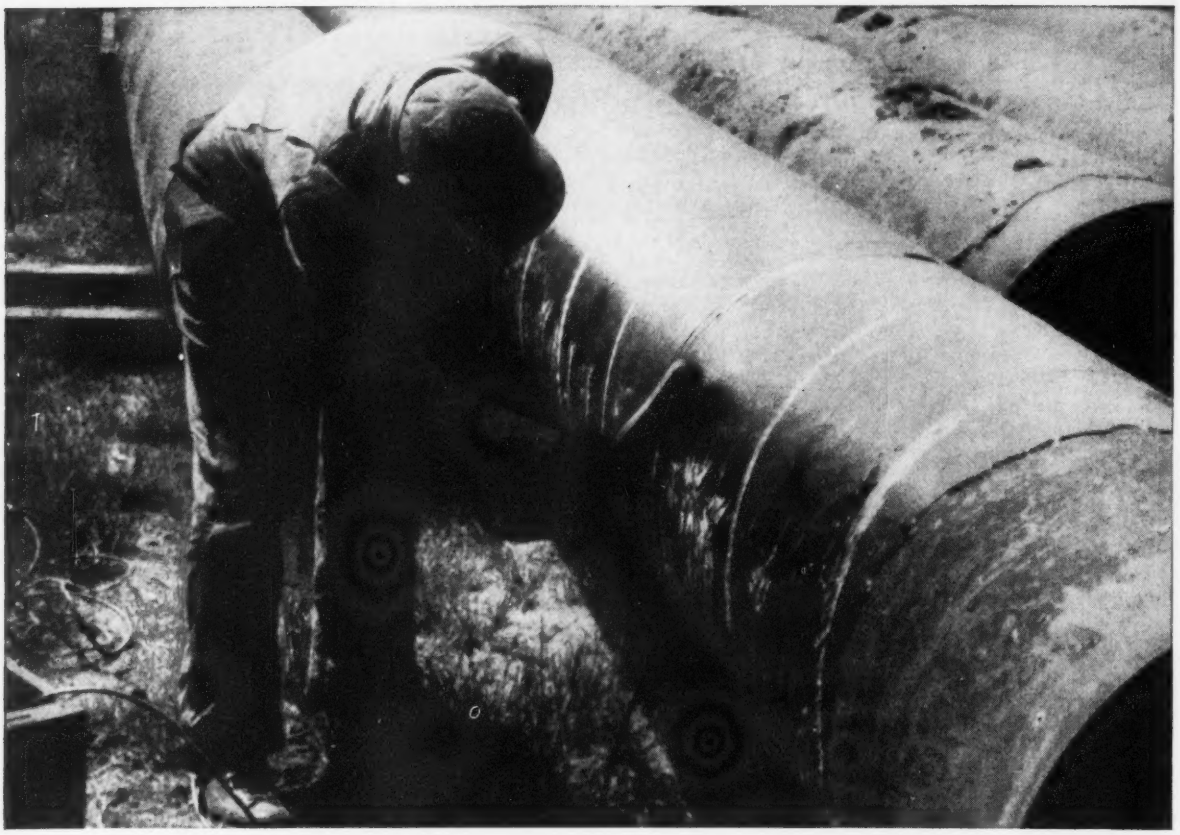
Members of many other great national associations that have met in San Francisco tell us that they have greatly enjoyed their conventions here and have found San Francisco an ideal place, both for working sessions and for recreation and entertainment. We are confident that you will have a similar experience and a record-breaking attendance.

With all good wishes,

Cordially yours,

George Christopher
Mayor

Now is the time to plan attendance at the 14th Annual NACE Conference and Exhibition



Natural Gas Pipeline Company of America Finds GAS LINE LIKE NEW AFTER 27 YEARS!

Bitumastic coal-tar enameled pipe to be re-used

In relocating its line to make way for a new highway near Truro, Iowa, the Natural Gas Pipeline Company of America removed some 900 feet of Bitumastic® enameled pipe laid in 1930. According to Mr. D. C. Allen, District Superintendent, this 24-inch seamless steel tubing is in such good condition that it will be reconditioned and used again as new pipe.

Applied by the rolling and "granny-rag" method in 1930, the Bitumastic XXH Enamel on this line was in excellent condition. The asbestos-felt overwrap, originally applied loose, was torn from soil stresses, but the Bitumastic enamel coating showed no serious disbonding or deterioration. At the time of relocation, the section had been under cathodic protection for two years. Natural Gas Pipeline Company of America records show no corrosion leaks on this section during the entire 27-year period underground.

Case histories such as this from prominent pipeline companies prove the unmatched protective ability of coal-tar enamel. In these days of rising material costs, you can't afford to gamble your entire pipeline investment on a less effective coating.

Your Koppers representative will be glad to give you full information on the superior protection of Bitumastic coal-tar enamels. Koppers District Offices: Boston, Chicago, Los Angeles, New York, Pittsburgh, and Woodward (Birmingham), Alabama.

KOPPERS

BITUMASTIC
REG. U. S. PAT. OFF.
PROTECTIVE COATINGS

INDUSTRIAL CLASSIFIED DIRECTORY

SAMPLE DRAFT

This is not intended to be a complete industrial classified directory in the corrosion field. It is a sample of the type directory that might be prepared if enough interest is shown. Companies listed were selected from those who advertised in CORROSION during the period January, 1950 through December, 1956.

APPLICATORS

Hill Hubbell & Co., Div. of General Paint Corp.
Mayes Bros.
Standard Pipeprotection, Inc.

ASBESTOS and ASBESTOS MATERIALS

Johns-Manville Sales Corp.

ASPHALT OR ASPHALTUM

Gulf States Asphalt Co.
Kerr McGee Industries
The Tapecoat Company

CABLE

Anaconda Wire & Cable Co.
Cathodic Protection Service

CASTINGS

ALLOY
Sandusky Foundry & Machine Co.
ALUMINUM
Aluminum Co. of America
Kaiser Aluminum Company
Reynolds Aluminum Co.
IRON
Duriron Company
LEAD
St. Joseph Lead Company
STEEL
Sandusky Foundry & Machine Co.
ZINC
St. Joseph Lead Company

CATHODIC PROTECTION

ANODES, ALUMINUM
Aluminum Company of America
ANODES, CARBON
National Carbon Company
Wholesale Coke Company
ANODES, GRAPHITE
National Carbon Company
Wholesale Coke Company
ANODES-MAGNESIUM
Apex Smelting Company
Cathodic Protection Service
Corrosion Services Inc.
Dow Chemical Company
Federated Metals, Div. of American Smelting & Refining Company
Royston Laboratories, Inc.
Standard Magnesium Corporation
Wholesale Coke Company
ANODES-NICKEL
International Nickel Company
ANODES-HIGH SILICON CAST IRON
Cathodic Protection Service
Duriron Company, Inc.
ANODES-ZINC
Cathodic Protection Service
Federated Metals Div. of American Smelting & Refining Company
GROUND BED-BACKFILL
Cathodic Protection Service
Wholesale Coke Company
ELECTRICAL SUPPLIERS
Cathodic Protection Service
Erico Products, Inc.
SHUNTS, COUPLING
American Steel & Wire, Div. of U. S. Steel Corp.
SPLICER
Minnesota Mining & Manufacturing Co.
RECTIFIERS
Cathodic Protection Service
Corrosion Rectifying Co.
Good-All Electric Mfg. Co., Electrical Mechanical Div.
Wholesale Coke Company

CEMENT

ACID & ALKALI RESISTANT
E. I. du Pont de Nemours Co., Inc.
Haveg Industries, Inc.
U. S. Stoneware Co.
ASBESTOS & MAGNESIA
Johns-Manville Sales Corp.

COATINGS

ACID & ALKALI RESISTANT—APPLICATORS
American Chemical Paint Company
Sline Industrial Painters
ACID & ALKALI RESISTANT—MANUFACTURERS
American Chemical Paint Company
Briner Paint Manufacturing Co.
Coast Paint & Lacquer Company
Cook Paint & Varnish Company
David E. Long Corporation
Dearborn Chemical Company
Inertol Company, Inc.
International Paint Co., Inc.
Kerr Chemicals, Inc.
Metal & Thermit Corporation
Mobay Chemical Company
Quelcor, Inc.
Steelcote Manufacturing Company
Tnemec Co., Inc.
Tropical Paint Company
U. S. Stoneware, Plastics & Synthetics Division
Wilbur & Williams Company
ALUMINUM
Tropical Paint Company
EPOXY TAR
Carboline Company
HOT DIP (GALVANIZING)
American Hot-Dip Galvanizers Association, Inc.
Nowery J. Smith Company
METALLIC SPRAYED
F. W. Gartner Company
NICKEL-ALLOY
Niphos Process Sales Corp.
PLASTICS
Amercoat Corporation
Bakelite Co., Div. of Union Carbide & Carbon Corp.
Gates Engineering Company
M. W. Kellogg Co., Chemical Mfg. Div.
Plastic Applicators, Inc.
Rubber & Plastics Compound Co., Inc.
Tropical Paint Company
Truscon Laboratories
United Chromium Div., Metal & Thermit Corp.
PLASTIC TAPE
Cathodic Protection Service
Dresser Manufacturing Div.
The Kendall Co., Polyken Sales Div.
Minnesota, Mining & Mfg. Co.
Plicoflex, Inc.
Royston Laboratories, Inc.
RUBBER
Hercules Powder Co., Inc.
Rubber & Plastics Compound Co.,Inc.
Tropical Paint Company
The Wilbur & Williams Company
ZINC
American Chemical Paint Company
Tar Products Div., Koppers Co., Inc.
The Wilbur & Williams Company

CHEMICAL MANUFACTURERS

Bryton Chemical Division
Columbia-Southern Chemical Corp.
Diamond Alkali Company
Mutual Chemical Div., Allied Chemical & Dye Corp.
Solvay Process Div., Allied Chemical & Dye Corp.

COAL TAR PRODUCTS

Barrett Div., Allied Chemical & Dye Corp.
S. D. Day Company
Mavor-Kelly Company
Pittsburgh Coke & Chemical Co. Protective Coatings Div.
Reilly Tar & Chemical Corp.
Tar Products Div., Koppers Co., Inc.
TAPE
Mavor-Kelly Company
The Tapecoat Company

ENGINEERING SERVICES

Cathodic Protection Service
Coreco Inc.
Corrosion Control Inc.
Corrosion Rectifying Company
Corrosion Service Ltd.
Ebasco Services, Inc.
Electro-Rustproofing Corp.
Harco Corporation
Hinchman Corporation
Huddleston Engineering Co.
Magna of Texas
D. Markoff Associates
Plastic Engineering Sales Co.
Francis W. Ringer Associates
Rio Engineering Company
Servicios Anti-Corrosivos, SA.
A. V. Smith Engineering Company
South Florida Test Service
Water Service Laboratories, Inc.

FABRICATORS

LEAD
Federated Metals, Div. of American Smelting & Refining Company
NICKEL
Haynes Stellite Co., Div. Union Carbide & Carbon Corp.
PLASTIC
Haveg Industries, Inc.
STEEL
Wickwire Spencer Steel Div., The Colorado Fuel & Iron Corp.
STEEL EQUIPMENT
Thornhill Craver Company
TITANIUM
E. I. du Pont de Nemours Co.

FITTINGS

GALVANIZED
Nowery J. Smith
LEAD
Federated Metals, Div. of American Smelting & Refining Co.
METAL
Bart Manufacturing Corp.
Aluminum Co. of America
Ampco Metals, Inc.
PLASTIC
Dresser Manufacturing Division
Haveg Industries, Inc.
Luzerne Rubber Company
May Company
Tube Turns Plastics, Inc.
RUBBER
The Luzerne Rubber Company

FLANGES

METAL
Ampco Metal, Inc.
PLASTICS
Tube Turns Plastics, Inc.

HOODS

METAL
Aluminum Co. of America

HOSE

PLASTIC
Resistoflex Corp.
U. S. Stoneware Co.
RUBBER
U. S. Rubber Company

INHIBITORS

American Chemical Paint Co.
Aquaness Dept., Atlas Powder Co.
Calgon Company
Cherokee Laboratories, Inc.
Commercial Solvents Corp.
Dearborn Chemical Company
Dowell Inc.
Geigy Chemical Corp.
Humble Oil & Refining Co.
R. S. Norris & Associates
Solvay Process Div., Allied Chemical & Dye Corp.
Tretolite Company
Universal Oil Products Co.
Visco Products Co., Inc.
Wright Chemical Corp.

INHIBITOR DUMP BAILER

Galloway Engineering Company

INSTRUMENTS

INDICATING, RECORDING
Cathodic Protection Service
M. C. Miller
TESTING, MEASURING
Branson Instruments, Inc.
Cathodic Protection Service
Crest Instrument Co.
Fisher Research Laboratory, Inc.
Labline, Inc.
M. C. Miller
Petroleum Instrument Co.
Research Laboratory, Inc.
Stewart Brothers, Div. of Instrument Laboratories
Tinker & Rasor
Wilkinson Products Co.
LABORATORY
Crest Instrument Company
Ebasco Services, Inc.
M. C. Miller

LABORATORY SERVICE

F. H. Maloney Company
Resin Research Laboratories, Inc.

LININGS

BRICK
LEAD
St. Joseph Lead Co.
PLASTIC
Amercoat Corp.
Coast Paint & Lacquer Co.
The Glidden Company
Humble Oil & Refining Co., Tech. Service Sales Dept.
M. W. Kellogg Co., Chem. Mfg. Div.
Metal & Thermit Corp.
Plastic Applicators, Inc.
Saran Lined Pipe Co.
Shell Chemical Corp.
Technical Service Sales Dept., Humble Oil & Refining Co.
Tube-Kote, Inc.
Tubular Lining Corp.
U. S. Stoneware Co.
RUBBER
Arco Rubber Processors, Div. of Automotive Rubber Co., Inc.

MASTICS

The Glidden Co.
Humble Oil & Refining Co.
Royston Laboratories, Inc.

METAL SUPPLIERS

ALUMINUM
Federated Metals, Div. of American Smelting & Refining Co.

(Continued on Page 116)

INDUSTRIAL CLASSIFIED DIRECTORY

METAL SUPPLIERS

(Continued)

BRASS AND BRONZE
Federated Metals, Div. of American Smelting & Refining Co.
Phelps Dodge Copper Products Corp.

COPPER
Federated Metals, Div. of American Smelting & Refining Co.
Phelps Dodge Copper Products Corp.

IRON
Colorado Fuel & Iron Corp.

LEAD
Federated Metals, Div. of American Smelting & Refining Co.
St. Joseph Lead Co.

MAGNESIUM
Standard Magnesium Corporation

MOLYBDENUM
Federated Metals, Div. of American Smelting & Refining Co.

NICKEL
The International Nickel Co., Inc.
Wickwire Spencer Steel Division, The Colorado Fuel & Iron Corp.

SILICON BRONZE
Armco Steel Corp.

STAINLESS STEEL
The International Nickel Co., Inc.
Lukens Steel Co.

STEEL
Lukens Steel Company
U. S. Steel Corp.
Wickwire Spencer Steel Div., The Colorado Fuel & Iron Corp.

TITANIUM
E. I. duPont De Nemours & Co., Inc.
U. S. Industrial Chemicals Co., Div. National Distillers Products Corp.

ZINC
Federated Metals, Div. of American Smelting & Refining Co.
St. Joseph Lead Company

ZIRCONIUM
Carborundum Metals Co. Div. of the Carborundum Co.
U. S. Industrial Chemicals Co., Div. National Distillers Products Corp.

PIPE

ALUMINUM
Aluminum Co. of America

ALUMINUM-BRONZE
Ampco Metal, Inc.

BRASS
American Brass Company

CAST IRON
American Cast Iron Pipe Co.

COATINGS
Plicoflex, Inc.
Saran Lined Pipe Co.

COPPER
American Brass Company

FIBRE
The Fibercast Corp.

PIPE

(Continued)

GALVANIZED
American Steel & Wire Company
U. S. Steel Corporation

HARD RUBBER
The Luzerne Rubber Co.

IRON AND SHEET
American Cast Iron Pipe Co.

LEAD
Federated Metals, Div. of American Smelting & Refining Co.
St. Joseph Lead Company

NICKEL
The International Nickel Co.

PLASTIC
Haveg Industries, Inc.
The Luzerne Rubber Co.
Tube Turns Plastics, Inc.

STAINLESS STEEL
Metals Processing Div. of Curtiss-Wright Corp.
Wickwire Spencer Steel Div., The Colorado Fuel & Iron Corp.

STEEL
Bart Manufacturing Corp.
Pipe Line Service Corp.

TITANIUM
E. I. du Pont de Nemours & Co., Inc.

WOOD
Michigan Pipe Company

WROUGHT IRON
A. M. Byers Co.

PIPE LINE INSULATOR

Brance-Krachy Co., Inc.
Cathodic Protection Service
Dearborn Chemical Co.
F. H. Maloney Co.
T. D. Williamson, Inc.

PLASTIC SUPPLIER

Diamond Alkali Company
M. W. Kellogg Co., Chemical Mfg. Div.

PORCELAIN SPECIALTIES

U. S. Stoneware Co.

PRIMERS

American-Marietta Co.
Carboline Company
Cook Paint & Varnish Co.
Inertol Co., Inc.
International Paint Co., Inc.
Royston Laboratories, Inc.
Rust-Oleum Corp.
Rust-Selle Company
Shawinigan Resins Corp.
Steelcote Manufacturing Co.
Tropical Paint Co.
U. S. Stoneware, Plastics and Synthetics Div.

PUMPS

The Duriron Co., Inc.
The International Nickel Co.
The Luzerne Rubber Co.
Texsteam Corp., Div. of Vapor Heating Corp.

RESINS

ACRYLIC
ALKYD
The Dow Chemical Co.
CASTINGS
CELLULOSE
EPOXY
Kerr Chemicals, Inc.
Shell Chemical Corp.
PHENOLIC
Bakelite Co., Div. of Union Carbide & Carbon Corp.
POLYETHYLENE
POLYESTER
American Cyanamid Co., Plastics & Resins Div.
POLYSTYRENE
VINYL
Bakelite Co., Div. of Union Carbide & Carbon Corp.
Shawinigan Resins Corp.

RUST PREVENTIVES

Solvay Process Div., Allied Chemical & Dye Corp.

SAND BLAST EQUIPMENT

Clementina Ltd.

SPRAY PAINTING EQUIPMENT

The DeVilbiss Co.
Spee-Flo, Hot Spray Heaters

STONEWARE

U. S. Stoneware Co.

STRAINERS

Thornhill-Craver Co.

TUBING

ALUMINUM
Aluminum Co. of America
BERYLLIUM COPPER
BRASS, BRONZE & COPPER
FIBRE
The Fibercast Corp.

TUBING

(Continued)

GLASS
GOLD
NICKEL
International Nickel Co.
PAPER
PLASTIC
U. S. Stoneware
PORCELAIN
RUBBER
SILVER
STAINLESS STEEL
STEEL
TITANIUM

VALVES

HARD FACED
LEAD
METAL
Alloy Steel Products Co.
Duriron Co., Inc.
International Nickel Co.
Texsteam Corp., Div. of Vapor Heating Corp.
PLASTICS
Haveg Industries, Inc.
Tube Turns Plastics, Inc.
HARD RUBBER
The Luzerne Rubber Company
TITANIUM
Pfaudler Company
ZIRCONIUM
Carborundum Metals Co., Div. of the Carborundum Company

YARD COATINGS

(See Applicators)

WRAPPERS— PIPE LINE COATINGS

ASBESTOS
Barrett Div., Allied Chemical & Dye Corp.
The Philip Carey Mfg. Co.
S. D. Day Company
Johns-Manville Corp.
Mavor-Kelly Co.
Midwestern Pipe Line Products Co.
Nicolet Industries, Inc.
Perrault Equipment Co.
Royston Laboratories, Inc.
Ruberoid Company
GLASS
The Philip Carey Mfg. Co.
Mavor-Kelly Company
Midwestern Pipe Line Products Co.
Perrault Equipment Co.
Royston Laboratories, Inc.

Would You Like to Have Your Company Name Listed in a Directory?

If you are interested in having your company name listed in a classified directory similar to the one above write before December 15 to A. B. Campbell, Executive Secretary, National Association of Corrosion Engineers, 1061 M & M Bldg., Houston 2, Texas indicating under which classifications (not limited to those printed above) you would like to have the name appear. A decision on publishing the directory will be made early next year if sufficient interest is shown in the project.

PROTECTION!

CHEROKEE LABORATORIES, Inc.

TUF-PLATE

CORROSION INHIBITOR

ADSORPTION!

WETTING!

RESULTS OF YEARS OF LABORATORY RESEARCH AND FIELD TESTING ARE NOW AVAILABLE TO THE PETROLEUM INDUSTRY

- OUTSTANDING CORROSION INHIBITORS FOR CRUDE PETROLEUM FLUIDS
- LONG LASTING EFFECTIVENESS
- EXCEPTIONAL WETTING AND ADSORPTION QUALITIES
- FAST POSITIVE FIELD SELECTION TESTS
- NEW FIELD TESTS TO GUIDE INHIBITOR SCHEDULES

CHEROKEE LABORATORIES, Inc.

MANUACTURERS OF TUF-PLUG, SUPER-PLUG, TEM-PLUG, AND CHEROKEE CYCLONE

HOME OFFICE AND MANUFACTURING PLANT IN TULSA, OKLAHOMA

Branches: Midland, Beaumont, Corpus Christi, Oklahoma City, New Orleans, Lafayette, La., Casper

TUF-PLATE INHIBITOR

New Ideas on Pipeline Coatings Aired at Houston

Cost Data Favor Hot Applied Systems, Panelist Asserts

Some new ways of thinking about pipeline coatings—and some unexpected information concerning the relative cost of alternative coating systems were presented at a meeting of Houston Section September 10. Interest in the panel discussion on underground pipe line coatings was evidenced by the attendance—in excess of 100. Representatives of coatings manufacturers and applicators in the Houston area were present in substantial numbers.

Tabulated data compiled by Lyle Sheppard, Shell Pipe Line Corp. based on a theoretical 200-mile pipeline indicated hot applied coal tar and asphalt coatings apparently enjoy a price advantage over plastic tape systems. Mr. Sheppard emphasized the figures were based on recent information from contractors, but did not necessarily represent a studied effort to establish relative prices of the systems considered.

Besides Mr. Sheppard, other panel members were E. R. Allen, Humble Pipe Line Company and W. H. Stewart, Sun Pipe Line Co., Beaumont, NACE president. O. W. Wade, Transcontinental Gas Pipe Line Co., who acted as moderator, also made a substantial contribution to the discussion. Mr. Stewart injected a note of caution by indicating that his 20 years' experience with pipe line coatings did not make it possible for him to determine which coatings were better than others.

Adhesion Is Tape's Problem

During the presentation by Mr. Sheppard on wrapping tapes, and in his subsequent remarks answering questions from the floor as well as remarks from other members of the panel it was conceded that a principal drawback of

(Continued on Page 120)

CORROSION ENGINEERING DIRECTORY

cathodic protection service
CPS
Twenty cathodic protection engineers with a combined total of over 150 years experience available to serve you with ABILITY and INTEGRITY.
Houston, 4601 Stanford St.
BRANCH OFFICES
Tulsa • New Orleans • Corpus Christi • Denver

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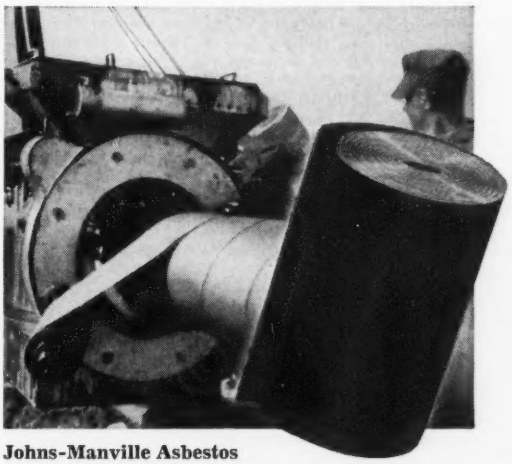
Johns-Manville Asbestos Wrap shields oil and gas pipeline coatings



Johns-Manville Asbestos Felt is available in 3 types for field application or mill wrapping:

#15 Asbestos Felt—the high-strength, uniform asbestos wrap that has given superior protection to pipe coatings for more than 30 years—under the most severe soil conditions.

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J-M Asbestos Wrap provides the strong protection a strong coating deserves and needs. It offers the most effective single protection against damage—prolongs the working life of the pipeline coating. Here's why:

Asbestos is an ageless mineral . . . the fibres are strong and tough—cannot rot or decay. As used in J-M Wrap the asbestos fibres are felted, then impregnated with a cold tar or asphalt saturant to form literally a flexible covering of stone. Like the "dillo's" tough shell that shields him so well—the enduring, "stonelike" J-M Asbestos Wrap shields pipeline enamels from earth loads, soil stress, and other forces that weaken coatings and permit corrosion of the pipeline.

Invite one of our engineers to show you the results of the three-year, extensive test program on "Pipe Line Coatings and Wrappers." Write to Johns-Manville, Pipe Division, 22 East 40th Street, New York 16, N. Y.

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REDUCES HAZARD • INCREASES SAFETY

Johns-Manville PRODUCTS FOR PIPELINE PROTECTION

Industrial Classified Directory Is Published

Included in this issue of CORROSION, beginning on Page 115 is a sample of a proposed Industrial Classified Directory. The directory is published to show the extent and nature of the indexing arrangement and to give an example of how such a directory will look.

Publication of such a directory is under consideration if sufficient interest is shown by companies seeking listings. Companies interested in listing their names in a proposed directory are invited to write to A. B. Campbell, Executive Secretary, National Association of Corrosion Engineers, 1061 M & M Building, Houston 2, Texas indicating under which classifications they would like to have their company's name appear.

Expressions of interest will be accepted through December 15, after which date a decision will be made as to whether or not the directory will be offered, charges for listings and circulation.

Cleveland Section Meets October 15 to Hear Tator

Cleveland Section meets October 15 at Manger Hotel, Cleveland to hear Kenneth Tator, K. Tator Associates, coatings consultant. Eight meetings beginning with a September 17 field trip to Sherwin-Williams Company have been planned.

Attend PERMIAN BASIN CORROSION TOUR
October 23, 24 and 25

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Other meetings scheduled and tentative programs are as follows:

November 19—L. West, Pipe Line Division, Standard Oil Co., Cleveland.

December 17—Anton deS. Brasunas, American Society for Metals.

January 21—Joint meeting with The Electrochemical Society, F. L. LaQue, International Nickel Co., Inc.

February 17—Matthew M. Braidech, National Board of Fire Underwriters.

April 15—J. J. Harwood, U. S. Naval Research Laboratory, Washington, D. C.

May 20—F. J. Ploederl, Wisconsin Protective Coating Co.

Nine Meetings Set by N. O.-Baton Rouge Section

New Orleans-Baton Rouge Section will hold nine meetings September 1957 through May, 1958. All meetings will be held at the Engineers Club, De Soto Hotel, New Orleans.

J. P. Barrett, Pan American Petroleum Corp., Tulsa, Okla. will be the featured technical speaker at the October 28 meeting. Barney Goodman, Tube Kote, Inc., Houston was scheduled on the September 6 meeting agenda.

Other meetings will be held as follows:

November 25—Hal Hynes, Mavor-Kelly Co., Houston.

Technical programs have not been announced for meetings to be held on December 30, January 27, February 24, March 24, April 28 and May 26.

New Ideas on—

(Continued From Page 118)

tapes, insofar as application to underground piping is concerned, is the problem of adhesion. Mr. Sheppard showed samples of several types of tapes, including those with integral adhesives and those which require separate application of primers.

Mr. Sheppard said, in connection with the problem of adhesion, that holidays in tape coatings frequently admitted water or other materials to destroy the adhesion of a considerable area of tape adjacent to the holiday. Cathodic protection applied to this pipe in an effort to offset the damage from the holiday, apparently would be effective only on the area represented by the holiday, while the vastly larger area of lost adhesion would be unaffected. This is the consequence of the high dielectric strength of the tape, he pointed out.

It was agreed by Mr. Sheppard, and concurred in with only minor objections from the floor, that a principal use of plastic tape coatings was in protecting short sections of pipe, or repaired areas. In this use it has obvious advantages over coatings applied hot, Mr. Allen said, in that a repair crew applies a coating of known dielectric characteristics, with reasonable ease, which is flexible enough to cover fittings, bends and irregularities.

Thin Films Have Promise

Application of thin resin films to pipelines for underground service is relatively recent, as pointed out by Mr. Allen, in discussing new developments in pipe line coatings. Most interesting of these films are the new coal tar-epoxy coatings, available on two or three-part systems for catalytic setting. In using them, he said, the effect of temperature was paramount. High temperatures reduce pot life materially, while at some low temperatures the films may not set at all.

Significant factors in considering thin film resin coatings outlined by Mr. Allen were:

1. High cost is drawback.
2. Good surface preparation is required. Multiple passes are required to get 25 to 30 mil coats.
3. Application at pipe mills on a large scale may make the materials competitive with conventional coatings.
4. Even mill coated pipe has the disadvantage of requiring field protection of weld gaps.

Recent development of a thermosetting resin for pipe coating which can be cured by passing a flame over it was reported by Mr. Allen. One advantage, he said, was that field joint coating could be applied readily.

Current Asphalts Reported Good

Mr. Wade, who discussed a wide variety of coatings, including coal tars, tapes, asphalts, concrete, mastic-concrete and others was commendatory on the merits of currently produced asphalt coatings. Asserting that more than 90 percent of the pipe under his supervision was being coated with asphalt, he said the quality has improved materially since it was recognized that asphalt from some sources was superior to that from others.

Early asphalts frequently showed poor performance, he said, although other installations in which asphalt was poured into a box built around a pipe are still in good condition. Economics of asphalt coatings is good now and he expressed a conviction that their quality was equal to coal tar.

Answering a question from the floor, Mr. Wade said a recent 2½ mile test section of pipe coated outside with an epoxy-modified coal tar was purposely handled as bare pipe in installation to see what the results would be. While it is too soon to be certain, he said, it is possible that the test will show favorable results. Recent improvements in the epoxies may make this type of coating even more satisfactory, he indicated.

Weighted Coatings Problem

In discussing weighted coatings, Mr. Wade said he believed Somastic coatings have given excellent results in submerged pipe. The recently developed Timcoat also seems to have promise besides having the advantage of being applied as a continuous coating and not requiring field joint protection.

Reinforced concrete coatings applied for negative buoyancy, Mr. Sheppard said, may have the disadvantage of having cathodic protection current shielded by the reinforcing steel.

Somastic coatings usually require more current for effective cathodic protection than coal tar, Mr. Sheppard said, but noted that the demand was uniformly distributed over the pipe area, while coatings of higher dielectric strength often did not give as good distribution.

Panel members agreed that coatings which resulted in a reduction in the number of times pipe required handling can be expected to show good cost characteristics. The fact that taped pipe can be lowered into the ditch immediately after wrapping was considered to be one of the principal assets of this type of protective film.

GENERAL NEWS

BOOK NEWS

Major Activities in the Atomic Energy Programs. 257 pages, 5¾ x 9 inches, paper. 1957. Superintendent of Documents, United States Government Printing Office, Washington 25, D. C. Per copy, $.75.

The semi-annual report of the Atomic Energy Commission to the Congress of the United States.

Index of Technical Articles. 102 pages, 8½ x 11 inches, paper. No. 3, April, 1957. Iota Services, Ltd., 38 Farrington St., London E.C.4. Subscription price, per 12 monthly issues, 6 guineas.

This issue includes a classified list of technical articles published during March, 1957 in about 500 technical periodicals in Great Britain, including 10 daily newspapers. The references consist of the title, a brief abstract and the necessary locator information.

A well-organized index is published both in numerical sequence and in alphabetical order. Abstracts are arranged in numerical sequence according to the index and are easily located. There is an alphabetical author index.

Uber die Aushartung von Aluminum-Kupfer Legierungen. (In German) by Von Dr. Harmann Franz. 156 pages, 6 x 9 inches, cloth. Aluminum-Verlag GMBH, Dusseldorf, Germany. Per copy, DM20.

Experimental results are given of examinations by X-ray, microscopic, mechanical, electrical and magnetic, calorimetric and other means of aluminum-copper alloys. Theoretical discussion covers thermodynamics and kinetics.

Passivierende Filme und Deckschichten (Anlaufshicten) Mechamismus ihrer-Anstehung und Ihre Schutzwirkung gegen Korrosion. (In German) Edited by H. Fisher, K. Hauffe, W. Wiederholt. 1956. 400 pages, 6 x 9 inches, cloth. Springer-Verlag, Reichpietschufer 20 Berlin, Germany. Per copy, DM 43.50.

Consists of the papers presented by 22 authors at a discussion on corrosion prevention at a session of Deutsches Ges. fur Metallkunde, at Frankfurt am Main, Oct. 13-14, 1955. The papers pertain to the following topics among others, oxidation, radioactive isotopes in studying high temperature oxidation, molybdenum oxidation, electrical and electrochemical aspects of films, mechanism of electrochemical passivation, mechanism of inhibition, experimental methods for investigation of metal oxidation, atmospheric corrosion of metals, corrosion of iron by steam, coatings from electrolytic polishing.

References are numerous and complete. Illustrations, charts and tabulated data are given. There is an extensive alphabetical subject index and an index to authors mentioned in the text.

Chemistry Research 1956. 86 pages, 6 x 9½ inches, paper. 1957. Report of the Chemistry Research Board with the Report of the Director of the Chemical Research Laboratory, Dept. of Scientific and Industrial Research, London. British Information Services, 45 Rockefeller Plaza, New York 20, N.Y. Per copy, 77¢

A wide variety of information on the work of British governmental agencies concerned. Principal interest for corrosion workers is in sections relating to metals corrosion where activities in connection with metals underground and exposed to various atmospheric and chemical environments are reported.

Details of other research work are given in general terms, including some information of high temperature oxidation, purification of elements and air pollution. Interesting information is given on the National Collection of Industrial Bacteria, which seeks to catalog non-medical bacteria of industrial importance. Studies on sulfate-reducing bacteria are described including means used to control their growth.

A list of publications and patents is included.

Engineering Properties and Applications of Plastics. By Gilbert Ford Kinney. 7 August 1957. 278 pages, 6 x 9 inches, cloth. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N.Y. Per Copy, $6.75.

A comprehensive treatise on the common plastic materials of construction. Chapters are given on polyethylene, polyvinyl chloride, polystyrene, acrylics, other vinyl-types, cellulose, phenolics, amino plastics, polyesters, polyamides and epoxies, silicones, rubbers.

An explanation is given of molding methods, engineering properties, thermal properties, optical properties, electrical properties. Extensive data are given on plastics chemistry with diagrams of typical molecules, potential energy, polymerization mechanics, aromatic hydrocarbons, acids, esters, anhydrides and particle-size distribution. Tables of properties of representative plastics are given. There is an alphabetical subject index.

Symposium on Industrial Water and Industrial Waste Water. 52 pages, 6 x 9 inches, hard paper cover. ASTM Spec. Pub. No. 207. June, 1957. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy, $2.

Six papers presented at the Second Pacific Area National Meeting, Los Angeles, Sept. 20, 1956 under auspices of ASTM Committee D-19 on Industrial Water. The papers discuss questions concerning probable water demand in Southern California for all purposes, including industrial; procedural methods used in controlling waste water disposal; sea water condensation units on shipboard producing fresh water, organic flocculants in water treatment systems.

Corrosion aspects of some of the subjects are considered briefly.

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Indian Institute's Technical Meeting Is In December

The Eleventh Annual Technical Session of the Indian Institute of Metals will be held in Bombay in December. The program includes a symposium on rare metals, the fourth series of educational lectures on phase transformations of steel and the fourth annual metallographic contest and exhibition.

NACE members in India at the time are invited to attend.

Urethane Data Included in Plastics Engineers' Papers

Some data on urethanes interesting to corrosion engineers was included in the meeting October 8 of Upper Midwest Section of the Society of Plastics Engineers, Inc. held at Minneapolis.

Two of the papers presented were:

Recent Advances in Urethane Materials, C. H. Wilson, W. L. Riedeman, University of Notre Dame.

Storage Characteristics of Polyurethane Resin, Joseph C. Fuono, U. S. Naval Powder Factory, Indian Head, Md.

Epoxy-Amine Systems Topic of FPVPC Paper

Among the numerous papers scheduled for presentation during the October 30-November 2 meeting of the Federation of Paint and Varnish Product on Clubs at Bellevue-Stratford Hotel, Philadelphia is:

Solvent-Free Epoxy Amine Systems, G. H. Ott, Ciba, Ltd., Basle, Switz.

CERTIFICATES of MEMBERSHIP in NACE

Certificates of membership in the National Association of Corrosion Engineers will be issued on request at $2 each, remittance in advance. The certificates, which measure 5½ x 8½ inches, are signed by the president and executive secretary of the association.

CERTIFICATES for PAST CHAIRMEN of REGIONS and SECTIONS

Certificates measuring 9 x 12 inches in size, prepared from an engraved plate, are available for issuance to regional and sectional chairmen. They will be supplied on request of the region or section at $7.50 each to be paid by the region or section, the cost to be classified as a non-reimbursable expenditure.

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A. B. Campbell, Executive Secretary

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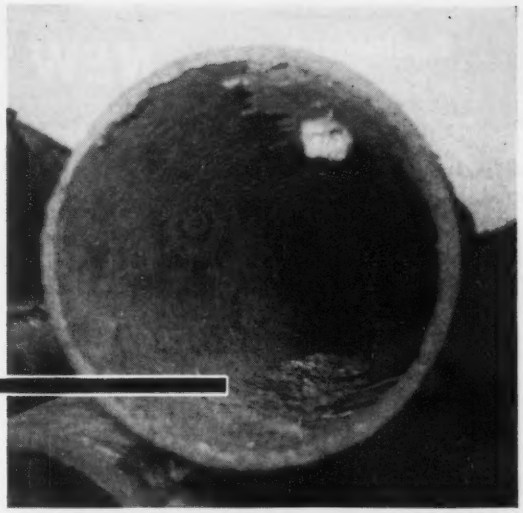

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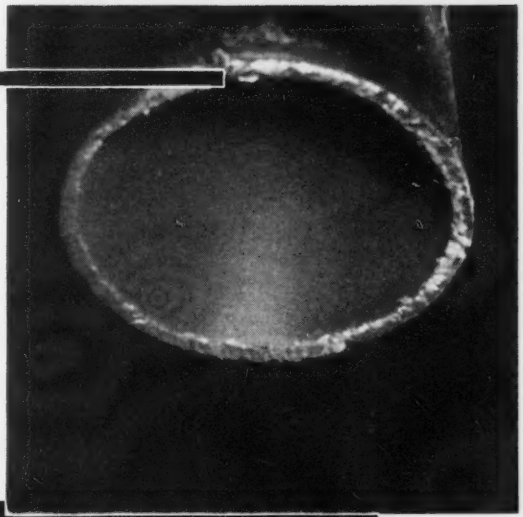

TO THIS

This smooth interior surface means improved "C" factor, restored throughput.

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SEE WHAT KONTOL 77 DID FOR THIS PIPE LINE

	Date	"C" Factor	Use of Pigs
BEFORE KONTOL 77	May	125	4 knife scrapers, 1 brush scraper
	June	125	10 knife scrapers, 1 brush scraper
	July	127	6 knife scrapers
	August	130	20 knife scrapers
	September	132	26 knife scrapers
	October	138	18 knife scrapers
KONTOL 77 WAS USED HERE!	December	141	14 knife scrapers
	February	141	6 knife scrapers
	February (following year)	141	**NO SCRAPERS FOR ENTIRE YEAR!**

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Aluminum Powder Metallurgy products commercially available now in the form of extruded shapes, forgings, sheet, foil, drawn and extruded tube, impact extrusions, fasteners and wire can withstand temperatures up to 900 F, according to Aluminum Company of America. They consist of unalloyed aluminum powder, each flake coated with aluminum oxide, which when compacted and worked produces a material far stronger and more stable than aluminum alloys produced by conventional methods. Three alloys currently are available.

Batteries

Nickel-Cadmium Button Cell batteries measuring 1⅜ inch by 3/16-inch for 250 milliampere hours and the same diameter and 5/16-inch thick for 500 milliampere hours of energy are being produced by Gulton Industries, Inc., 212 Durham Ave., Metuchen, N. J. They are rechargeable.

Coatings

Temploc, pipe insulated by a layer of glass fibers and covered by a spirally-wound jacket of glass cloth and polyester resin is made by Temploc, Inc., Baldwin Park, Cal. It is designed for underground transportation of heated liquids.

•

No. 42 Deadener, an insulating coating that can be applied at temperatures from 40 to 70 F will give corrosion protection and dampen noises from steel surfaces. It is a product of Philip Carey Mfg. Co., Lockland, Ohio. A descriptive booklet is available on request.

Metals-Ferrous

Micro-Rold 430, a nickel-free, low carbon, ferritic steel with a 17 percent chromium content is described in an illustrated 24-page and cover booklet available from Washington Steel Corp., Washington, Pa. The booklet illustrates many uses, give analysis and physical properties and lists resistance to numerous corrosives.

•

Tube-Turn stainless welding elbows, tees, reducers and flanges were used in fabricating the first sodium cooled, graphite-moderated thermal nuclear power reactor system for generating electricity. The experimental installation, being conducted for the Atomic Energy Commission by Atomics International, includes a 20,000 thermal kw reactor. California Edison Company has constructed a 7500 kw steam generating plant at the experiment site, Santa Susana, Cal. which will use reactor heat to power turbines. The installation is the prototype of proposed 75,000 kw generating plants, one of which is now in negotiation with Consumers Public Power District of Nebraska.

Instruments

Union Carbide Nuclear Company and Union Carbide Ore Co. are undertaking research with a million-volt van de Graaf accelerator at Sterling Forest, N.Y. Properties of various materials bombarded by high-energy particles from the accelerator are being studied.

•

Dental Instruments and supplies, consisting of plastic boxes, rubber gloves, rubber and plastic tubing, abrasive wheels, burs and other dental items with industrial applications can be obtained from Industrial Products Div., Orthodontic Specialty Laboratory, 1827 Spruce St., Philadelphia 3, Pa. A 24-page catalog is available.

•

University of Arkansas soon will receive the first commercial Cockroff-Walton

(Continued on Page 126)

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Power-Driven INJECTORS

Here is a complete new series of pumps, designed to give greatest accuracy and economy in chemical injection. **Check these features:**

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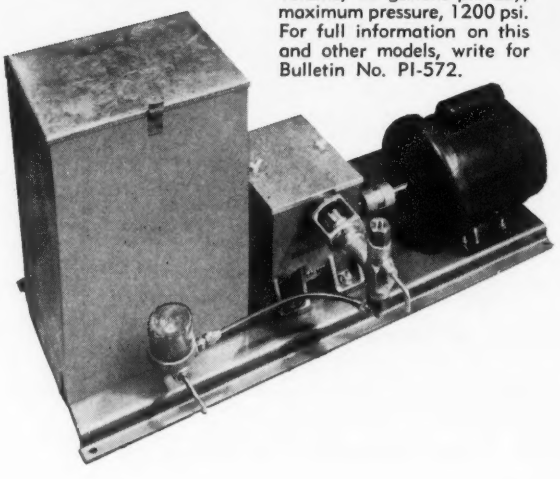

Model SAC-4001 Injector. Piston size, ¼"; maximum volume, 10 gallons per day; maximum pressure, 1200 psi. For full information on this and other models, write for Bulletin No. PI-572.

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Humble internal pipe coatings are also available in vinyl and phenolic formulations.

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NEW PRODUCTS

(Continued From Page 124)

Accelerator made in the United States by Applied Radiation Corp., Walnut Creek, Cal. The 350,000-volt electron volt machine will accelerate a 400 microampere beam of positive ions to generate neutrons of various energies for nuclear physics and chemistry research. Up to a hundred billion neutrons per second can be produced. It was given the university by AEC.

•

Sonizon SO-200 portable ultrasonic thickness testers can determine metal thickness from one side with an accuracy of plus or minus 3 percent on thicknesses between .027-inch and 4 inches. The battery-operated machine is available from Magnaflux Corp., 7300 West Lawrence Ave., Chicago 31, Ill.

Plastics

Resistoflex Corp., Roseland, N. J. is making fluorocarbon laboratory beakers in 250,500 and 1000 cc sizes. They are non-porous and have low interfacial tension, reducing cleaning time.

Welding

EB Weld Inserts, manufactured by Arcos Corp., Philadelphia, permit welding from one side only such vessels as air tanks capable of withstanding air pressures of 3000 psi. The inserts are fused with automatic inert gas shielded tungsten arc equipment.

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MEN in the NEWS

Harry S. Ritterson is now sales engineer for Gates Engineering and Delaware Valley Steel Works, a Gates subsidiary.

•

Harold Hessing has rejoined the chemical metallurgy section and **August E. Dauses** of the Shops Division has been awarded the Department of Commerce Silver Medal for Meritorious Service at the National Bureau of Standards, Washington.

•

Oscar T. Markze has been appointed vice-president, fundamental research, United States Steel Corp. He was formerly director of research, U. S. Naval Research Laboratory.

•

Ralph S. Jessup, an authority on heat standards at the National Bureau of Standards has been awarded the Department of Commerce Gold Medal for Exceptional Service, recognizing his work in the bureau over 40 years.

•

Leason H. Adams, formerly a director of the Geophysical Laboratory at Carnegie Institution, Washington has been appointed a consultant to the director of the National Bureau of Standards on high pressure measurements and standards.

•

K. G. Compton and **A. Mendizza,** Bell Telephone Laboratories, Murray Hill, N. J. have been awarded the Sam Tour Award by ASTM for their paper "Galvanic Couple Studies by Means of the Threaded Bolt and Nut Wire Test."

•

Robert E. Galer, retired brigadier general of the United States Marine Corps has joined Temco Aviation Corp. as manager of engineering programs. He formerly was director of the Guided Missiles Division of the Navy Bureau of Aeronautics.

•

E. G. Bailey, formerly president of the American Society of Mechanical Engineers will be honored with a life membership in recognition of his long service to the society and work in engineering education.

•

Kempton H. Roll has been appointed full time executive secretary and treasurer of the Metal Powder Association.

•

Morton M. Jenkins, formerly chief metallurgist for Pittsburgh Forgings Company has been named Steel Sales Metallurgist for A. M. Byers Co. in connection with the firm's expanded activity in the stainless and specialty steel business.

•

Charles H. Topping, senior architectural and civil consultant for E. I. du Pont de Nemours & Co. Inc. has been elected president of the Building Research Institute.

•

Basil T. Lanphier, NACE member has been named manager of research for Carpenter Steel Co., Reading, Pa. **Howard O. Beaver,** has been made production metallurgist in charge of melting and hot working.

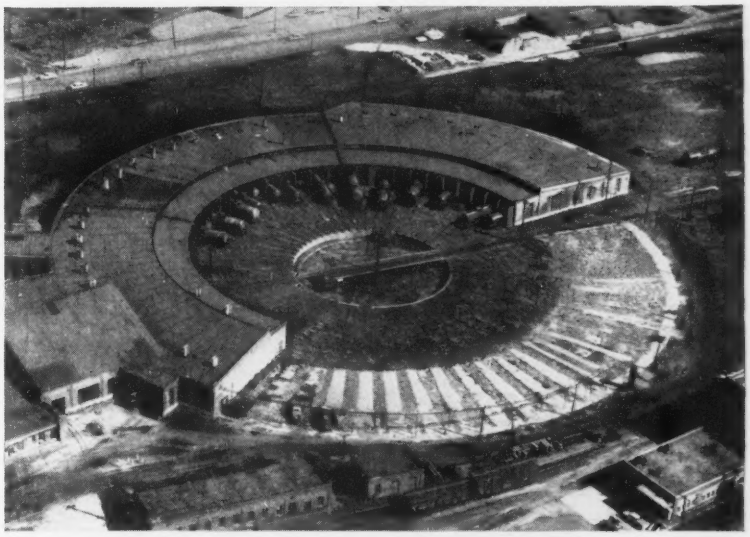
(Continued on Page 128)

UNICHROME Trade Mark

News about
COATINGS for METALS
Metallic.....Organic.....Decorative.....Protective

Phenolic linings improved for tank use

Unichrome Series B-124 Linings give reliable protection — are now being applied by specialists



Ingenuity! Lithcote Corp., a prominent applicator of Unichrome Phenolic Coatings, has adapted a former railroad roundhouse to line tank cars. Cars are sprayed, switched to the baking facilities, and can then be sent rolling back to the customer in short order.

Phenolic-type coatings, the acknowledged "workhorse" for tank lining applications, have a long and successful record in corrosive service. They'll be specified even more now that two problems have been overcome by Metal & Thermit.

(1) Unichrome Phenolic Coatings have been developed that assure more uniform linings, greater impact resistance, and improved protection. (2) Experienced, independent applicators across the country now make it possible to ship large equipment to the nearest specialist, get fast service, save on transportation.

UNIQUE PHENOLIC COATINGS

Several pigmented materials in the Unichrome B-124 Coatings group give 2-mil thicknesses per dry coat. This is twice that of ordinary phenolic coatings. Fewer coats are needed. Clear coatings in the group bake into a hard, smooth film for an easy-to-clean top coat.

BUILT-IN CONTROL

Unichrome Coating B-124-17 is a widely specified phenolic formulation that is gray when wet, olive drab when fully cured. This cure-control offers reliable visual inspection of a job well done. It is valuable for avoiding undercuring in large tanks and tank cars due to uneven heat application.

Details on these Unichrome Linings are given in Bulletin Chem-C-2. Contact Metal & Thermit also for names of nearest applicators of Unichrome Series B-124 Linings.

Reports show Unichrome Plastisols give record service

Unichrome Plastisols, which produce thick vinyl coatings, are withstanding conditions that rapidly deteriorate unprotected metals or other materials. To illustrate:

Plastisol coated ducts exhausting strong acid fumes show no deterioration after 4 years.

After 6 months service, plastisol coated agitators for chemical mixers show absolutely no wear. Rubber coatings, used previously, started to fail after only one month.

Plastisol coating on a 5-ft. bubble tray at bottom of a 60-ft. scrubbing tower has been withstanding acid fumes and caustic solutions for over 2 years now.

A report shows Unichrome Plastisol Coating on racks withstood 6 years of daily use in corrosive plating and cleaning solutions.

APPLICATION NO PROBLEM

Unichrome "Super 5300" Coating spray-applies seamless films as thick as sheet linings. It delivers up to 60 mils per coat. "Series 4000" Plastisols apply by dipping, slushing, troweling or other convenient means. Send for Bulletin VP-2 or name of the nearest applicator of Unichrome Plastisols.

Unichrome is a trademark of Metal & Thermit Corp.

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In Canada: Metal & Thermit—United Chromium of Canada, Limited, Rexdale, Ont.

Paint guards steel building

against corrosive fumes

With RUSTARMOR® system — a rugged Inertol® long-oil alkyd resin enamel and primer—the exterior of an all-steel fertilizer mixing plant has been shielded against metal-corroding acid fumes. Thanks to RUSTARMOR, this plant —operated by Lincoln Service & Supply Co., Grand Island, Nebraska — **has for four years enjoyed maximum protection with no upkeep expense.**

The RUSTARMOR system is especially designed to protect exposed metal surfaces against rust, corrosion and all atmospheric conditions. It's tough and long-lasting. And RUSTARMOR never turns sticky, because it's hygroscopically controlled. Available in dozens of glossy weather- and fade-resistant colors that protect your plant and keep it attractive for long, low-cost years.

For more facts, get informative bulletin and color card C560. Simply write your name, title, firm name, address in margin of this page and mail.

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is Your Guarantee of

PREVENTIVE MAINTENANCE

For Pipe, Pipe Joints, Fittings, Couplings, Tanks, Tie Rods and Conduit

To reduce maintenance costs, get the best possible protection at the lowest possible cost. Since 1941, TAPECOAT has proved its superiority in combating corrosion. This quality coal tar coating in handy tape form is self-bonding—simple to apply with the use of a torch. Requires no trained help. Cuts maintenance and replacement costs. Comes in rolls of 2", 3", 4", 6", 18" and 24" widths. Also available in asphalt.

Write for brochure and prices

The TAPECOAT *Company*

Originators of Coal Tar Coating in Tape Form

1521 Lyons Street, Evanston, Illinois

MEN in the NEWS

(Continued From Page 126)

Albert P. Spooner, chief metallurgical engineer of the Steel Division of Bethlehem Steel Company and **Nevil Greenwell,** assistant to the manager of research of the company have retired. Mr. Spooner began working for the company in 1915 and Mr. Greenwell in 1913

•

Ralph R. Calaceto has been made manager and sales engineer of the Process Equipment Division of Automotive Rubber Co., Inc., New York City office.

•

Irving Langmuir, world renowned scientist and Nobel prize winner, who was on the staff of the General Electric Research Laboratory from 1909 until 1950 died August 16 from a coronary thrombosis.

•

Charles P. Siess, Jr. has been named assistant general sales manager of Tretolite Company. He has had over four years' experience with corrosion problems in Arkansas, North Louisiana, Mississippi and East Texas as well as at Maracaibo and Trinidad.

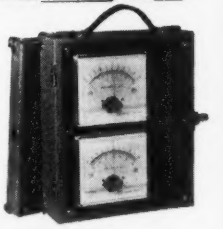
•

Ralph J. Rice is new sales representative of Plastic Applicators in its Lafayette, La. office. He has had previous experience in corrosion control with a leading oil company and with Tretolite Company.

Stewart

TEST EQUIPMENT

Locates Trouble FAST



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MILLIVOLTMETER, a low 2.5 Millivolt range . . . 2.5 to 500 mv in 6 ranges.

Oak Case, 9"x10"x4½" — Wt. 6 pounds.

STEWART BROTHERS

Division of Instrument Laboratories

Chicago 10, Illinois

TECHNICAL TOPICS

Capitalized Coating Costs Method Economical For Southeastern Pulp and Paper Mill Steel*

Abstract

A comparison is made between capitalizing coating costs for pulp and paper mills in the Southeastern part of the United States by sandblasting and pre-coating structural steel before erection with selected resin materials and the more common method of coating steel after erection and charging substantial recoating costs as a maintenance item. Savings in 10 years by using the capitalized cost method average $117,345 per 1000 tons after tax deductions.

A new standard of sandblasting is proposed which results in leaving adherent patches of scale not over 1-inch square and an over-all key pattern on the surface. **1.2.2**

IN 1949, a personal survey of twelve pulp and paper plants in Florida, Georgia and South Carolina indicated that between $200,000 and $250,000 a year was being spent per 300-ton-per-day plant on paint programs, material, labor and overhead. Two plants, built about that time, each used about 2000 tons of coated steel. This included structural shapes, plate, fabricated tanks, grating and the like. At the yearly cost found, a maintenance charge of 40-50 cents per square foot per year was being paid.

In 1949 paint programs were based on Northern experience, on the diligence of paint salesmen and on the individual initiative of plant paint foremen who were being worked to death in an attempt to stay ahead of corroding plant. High painting costs were accepted as a necessary evil and little effort was expended in trying to cut costs. Not until it was realized that conditions in the Southeast differed very radically from those in the North was progress made in mitigating corrosion and decreasing yearly maintenance costs.

Environments in the Southeast tend to show much higher corrosion rates than in sections of the country considerably to the north. Sustained humidities are much higher, ultra-violet ray exposure is more intense and high atmospheric temperatures of greater duration. Summer and autumn thunderstorms and their attendant heavy charges of lightning probably fix more nitrogen from the air and bring down enough nitric acid to weigh definitely in the corrosion balance. After the relatively greater severity of Southeast corrosion problems was recognized, it was possible to make great strides in mitigating corrosion and reducing the unit cost of plant maintenance. Some of these advances are:

Synthetics Prove Helpful

Synthetic-resin based materials have proved sufficiently resistant to weather and chemicals involved in processes to permit their useful life to be computed in terms of years rather than months. Establishing this fact had far reaching consequences all of which have not been fully evaluated. Most important result was a radical change in the attitude of plant painters who, up until this time, had been decorators-without-hope, machines for conveying color from a bucket to a surface on which it did not last from one shutdown to the next. Concurrent with acceptance of the new synthetics, pride of workmanship emerged, paint people generally sought knowledge about the products and methods of applying them and some of the better qualities of the synthetics themselves were strengthened by simple pride of the workman in his work.

As painter's tools improved, more material could be applied to greater areas in less time and with less effort. Roller coating, paint heating and pressure-fed brushes became accepted tools of the trade.

Surface Preparation Acceptance Gains

A long step toward lower costs came with the general acceptance of the fact that the day of miracles was over and there was no substitute for adequate preparation of surfaces.

Sandblasting is just now being accepted as the best and most effective method of surface preparation. Recognition is dawning that sandblasting is an art in itself, with highly developed techniques involving details such as machine sizes and types, nozzle diameters and shapes, hose diameters and shapes, blasting pressures and sand grain sizes and permissible variations. Incorrect sand sizing alone can increase the volume of coating used by an unnecessary 10-15%. Correct nozzle diameters and interior profiles will increase work rates from 50 to 100 percent.

Concurrently with new coating techniques changes were made in structural design to make corrosion control easier by substituting for corrosion-prone constructions those relatively resistant, by substitution of members whose surface exposed to corrosion was considerably less, inclusion of drains in blind, upward-facing pockets and seal-welding to prevent corrosion in areas inaccessible to cleaning and surface preparation.

Opinions on Economics Vary

A difference of opinion still exists among executive heads of pulp and paper mills as to whether it is less costly to do a first class protection job on an original installation and capitalize it, or do a decorating job on capital charge and later do a proper protection job with maintenance dollars as an expense item. This difference of opinion is elaborated at some length in this article.

Considerations of total money available for capital expenditure, of course, will influence decisions. However, given sufficient capital, to properly protect structure from the beginning is much less costly procedure than trying to do so after operation has begun, whether the cost of maintenance painting is considered an expense item or not.

Two Methods Are Compared

Comparing total costs of two methods of protecting a pulp and paper plant on a 10-year basis:

1. By shop priming all steel and recoating after erection with two coats of average good paint, to a total thickness of 2 mils. Or

2. By blast cleaning all incoming steel before erection, on site, under owner supervision and applying two or three coats of good synthetic coating to a thickness of 5 to 10 mils, depending on the corrosive nature in the area into which the steel is to go and the shape of the structure to be coated.

Details of First Method

Shop prime work by structural fabricators is notably of poor quality and shows little sign of improving. Fabricator's priming space is limited, speed in moving steel to cars is vital and nothing but nuisance value is assigned to effectiveness of this prime coat. In other words it is not the fabricator's problem.

The sequence of performance under Method 1 is approximately as follows:

1. Shop primed steel arrives on site and is erected. A contract painter generally is employed to apply the two coats of coating after erection. It often happens production executives are urging the paint contractor to clear the decks so operation of the plant can begin. The general result is that three coats of material are applied over an almost wholly unprepared surface. Sometimes the second and third coats will have been scamped either with too much thinner or with too little time to do a good job. The net result is that the plant is decorated with a color scheme approved by the company's directors, the contract painter can leave the site and what is considered most important, "Production" can get on with the job.

In three months plant fumes will have attacked the paint film on all weather-exposed steel and inside the plant, particularly in recovery, stock-washer and digester buildings, rust will be popping at welds and rivets, beam, channel and angle edges, pipe railings, steel gratings, etc. If within these three months Production has been able to solve primary problems and the plant is operating

(Continued on Page 130)

★A paper presented under the title "Corrosion Protection of Pulp and Paper Mill Steel in the Southeastern United States by Capital and/or Maintenance Dollars," by Henry T. Rudolf, Atlantic Coating Co., Inc., Jacksonville, Fla. at a meeting of Southeastern Region, National Association of Corrosion Engineers, Charlotte, N. C., November 8-9, 1956.

Capitalized Coating—

(Continued From Page 129)

close to capacity, attention then is turned to improving the appearance of the place and protecting it from further damage.

So far capital cost has been from three-quarters to two cents per square foot for shop priming and from six to eight cents per square foot, per coat, for the two cover coats, an average of 14.875¢ per square foot or about $37.18 per ton of coated steel.

Coating and corrosion control people are called in at this point to see what can be done and how much it will cost. The consensus may consist of a recommendation that five mils of any good protective coating should be applied over adequately prepared surfaces at a cost estimated at 30 to 40 cents per square foot or $87.50 per ton.

Problems With Surfaces Arise

It appears that adequate surface preparation includes sandblasting and this cannot be done in operating plant for obvious reasons. Equally obvious is the fact that the plant cannot shut down so the job can be done, nor will any future shutdown be of sufficient duration to permit it. The compromise then specifies all surfaces be thoroughly cleaned either by hand or power wire brushing followed by an inhibitive primer with special anchoring qualities; or, disc sanding of the surfaces followed by the same special primer.

Hand Cleaning is Tedious

To do a complete, thorough and conscientious job of hand surface cleaning on polluted surfaces in an operating pulp and paper mill is the meanest of mean jobs for an applicator because every foot of surface to be coated must be cleaned, with consequent cost of access equipment such as scaffolding, bosun chairs, walk-boards, ladders etc., plus high cost in time per unit area so cleaned.

Brush-blasting a polluted surface, if feasible, is faster, cheaper and more effective as surface preparation than any form of hand cleaning. A combination of commercial-blast on rusty areas and brush-blast on coated areas approximates in expense the cost of good hand cleaning but is less effective as surface preparation. So, for the purposes of this article consider the cost of sandblasting and good hand cleaning equal, although in practice good hand cleaning probably will be somewhat more expensive.

No reputable contractor or coatings manufacturer will guarantee the life of coating applied under these surface preparation conditions. Mill scale is liable to come free at any time, taking sheets of coating with it. Further, mill chemicals have soaked into the steel through areas of broken coating and these chemicals will react with vapor seeping through the new coating and drive it from the steel surface with corrosion products from beneath.

Maintenance Costs Mount

On the basis of experience it is reasonable to presume a good coating so applied might last from one to four years and it is also presumed that some 80 percent of the original coating would require refinishing. Taking the long estimate of four years life, every ton of steel so far has cost $37.18 originally plus 80 percent of $87.50 or $70.00 for corrosion protection. However the end is not in sight, because at the end of four years the remaining 20 percent of the original coating will require recoating at $87.50 and some 40 percent of the 80 percent first recoat will require a touchup. The remainder of the surface will receive one coat for appearance. This costs $87.50 per ton on 20 percent of surface, $60.00 per ton on 32 percent of surface and $25.00 per ton on the remaining 48 percent of surface.

At the end of four years then, each ton of steel has cost $37.18 in capital, and $70.00 plus $17.50 plus $19.20 plus $12.00 in maintenance funds. Projecting to ten years, it is not unreasonable to suppose that another $150 will be spent per ton from maintenance funds to protect the steel and maintenance appearance. Total cost at the end of ten years in capital and maintenance is $305.88 per ton of steel.

In actual practice this figure is affected by other considerations. If the plant hires a paint crew of sufficient size to keep up with current corrosion and cover large critical areas during shutdown periods, through overhead, fringe benefits and other charges this will cost about 15 percent more than the same work done by a painting contractor. Furthermore the capital cost must be amortized or written off in 20 years.

In ten years half the original capital charge of $37.18 ($18.59) must be subtracted from the total per ton cost and 6 percent interest on capital borrowed must be added. This brings to $305.88 the original capital cost and contract maintenance, minus half the original capital cost for write off, plus 15 percent of the contract maintenance cost as well (15% of $118.70) or $17.81, plus 10 years interest on borrowed capital, a gross figure of $327.40 per ton of steel.

Second Maintenance Method Described

Contrast the preceding with that of another way to do the work.

Deliver all steel to site without shop coat, sandblast all surfaces to be coated and apply on the ground the specified number of coats of material required in

(Continued on Page 132)

TABLE 1—Tabulation of Capital and Maintenance Coating Costs Per Ton of Steel

METHOD ONE						METHOD TWO				
ITEM	Original	During 1st Year	During 4th Year	During 4th Thru 10th Year	Total Expense End of 10th Year	ITEM	Original	End of 6 Years	End of 10 Years	Total Expense in 10 Years
Shop-coat plus 2 spray coats to 2 mils	(1) 37.18				37.18	Method 2. Blast and coat on ground to 5 mils	(1) 63.13			63.13
Complete clean and recoat with good Synthetic to 5 mils 80% of surface 87.50 x .80	(2)	70.00			70.00	Single spray color coat @ 22.50 ton	(2)	22.50		22.50
Complete clean and recoat with good Synthetic to 5 mils 20% of surface 87.50 x .20	(3)		17.50		17.50	Credit 2 unexpired years of 6 year life, covercoat 20.00 x ⅓	(3)		—7.71	—7.71
Retouch 40% of (2) surface @ 60.00/T	(4)		19.20		19.20	Spot clean and recoat to 5 mils, 5% of total @ 63.13/T	(4)	3.16		3.16
Single coat on 48% of surface @ 25.00 per ton 25.00 x .48	(5)		12.00		12.00	15% increase of cost for plant painters (2) + (3) + (4) x .15	(5)		2.69	2.69
General coating maintenace estimated	(6)			150.00	150.00	Credit for lack of shop coat	(6) —6.50			—6.50
15% increase of expense due employment of plant painters (2) + (3) + (4) + (5) x .15					17.81					
Write-off of 10 years of capital expense (period of 20 years) (1) x .5	(7)				—18.59	Write-off of 10 years capital expense (period of 20 years) (1) — (6) x .5	(7)		—28.32	—28.32
6% interest on $37.18 for 10 years	(8)				22.30	6% interest on $56.63 for 10 years	(8)			33.98
Total cost of capital and maintenance	37.18	70.00	48.70	150.00	327.40	Total cost, capital and maintenance	56.63	25.66	23.30	82.93
Cost per square foot, per year					0.131	Cost per square foot per year				0.03317

other Wrought Iron case against corrosion

58

YEARS OF SERVICE

Longitudinal photomicrograph S-64-2 (100x) shows a typical area—parallel to rolling—of the microstructure observed in the subject 5-inch wrought iron pipe.

Metallurgical Report 6628—Underground Oil Line*

This report deals with the metallurgical examination of a length of 5-inch wrought iron pipe, submitted to our laboratory for investigation and comment.

The sample submitted has been in service 58 years, yet found to be in excellent condition. Note that the scale is tightly adherent to the surface of the pipe.

ROCKWELL HARDNESS

A cross-sectional ring was prepared for hardness determination. Hardness values taken around the cross-sectional ring indicated a B66 hardness typical for wrought iron pipe and showed that the strength and ductility were likewise normal.

CHEMICAL ANALYSIS

Carbon—.018%; Manganese—.040%; Phosphorus —.236%; Sulphur—.019%; Silicon—.186%; Iron Silicate—3.54%.

SUMMARY

Results of the laboratory examination serve as positive identification of the subject sample as wrought iron. The excellent state of preservation of this sample after more than a half-century of service is indicative of wrought iron's longevity in corrosive service. A. M. Byers Company, Clark Building, Pittsburgh 22, Pennsylvania.

*Name of actual installation given on request.

Write for free cloth-bound book, *Wrought Iron: Its Manufacture, Characteristics and Applications.*

BYERS
WROUGHT IRON

Capitalized Coating—

(Continued From Page 130)

different plant locations before erecting the steel.

White or gray blast specifies the complete removal of all material foreign to a steel surface, including stain left behind removed mill scale. With a ⅜-inch cylindrical-inner-profile blasting nozzle, silica sand in excess of 90 percent SiO_2, sand grain size from 20 to 30 mesh, or closer 24 to 30, air pressure 90-100 psi at the blasting nozzle, on new plate steel waist-high to solid footing for the nozzle operator, a blasting rate of 90-100 square feet per hour can be achieved at a cost of 17-20¢ per square foot.

Commercial blasting specifies the complete removal of all mill scale, rust and rust scale, but does not remove adhesive mill scale or stain. Under the same conditions limiting white blast above, commercial blasting will show a blasting rate from 400-450 square feet per hour at a cost from 9-12¢ per square foot.

Brush blasting removes only loose material in the way of mill scale and rust, does not remove adhered mill scale, does remove old paint and does not pock the whole surface as a mechanical anchor for coating. An approximate blasting rate under the same limiting conditions as above should be between 1100-1300 square feet per hour at a cost from 3-5¢ per square foot.

Of the specifications above only white blast is acceptable for a coating to be immersed; commercial blast is widely accepted for non-immersed or weather exposed coating; brush blast is not strictly acceptable for either type of synthetic coating exposure because the risk of coating destruction from beneath is very high if mill scale in any area larger than a few square inches comes free from the mother material.

New Sandblasting Specification

A type of sandblasting called anchor blasting specifies removal of all rust and rust scale, old paint and other foreign material and leaves behind no mill scale larger than one square inch. To obtain this small residual scale area, loose scale will be driven off the surface and the small pieces of scale left inevitably will be pocked to anchor depth by edges of the blast pattern in cutting to small dimensions.

The purpose of this type of blasting is twofold:

1. The blasting rate is high, 600-800 square feet per hour and the cost is low, 5-6¢ per square foot, so the economy is obvious.

2. A good anchor for coating is secured without removing all mill scale and without the risk that a piece of scale coming free will break the coating. A piece of mill scale one foot square coming free will break the coating. A piece of mill scale 1 inch square coming free, is insufficient in weight or volume to break a 5-mil synthetic coating.

Cost of anchor blasting is 6¢ per square foot on the ground. The cost of a coating material (if no bleach areas are to be coated) should be about 9¢ per square foot for 5 mils and 13¢ for 10 mils of vinyl mastic. Application cost using conventional spray and heating equipment, will be that of one brush prime coat and two good heavy hot spray coats for 5 mils or one brush and one doublepass hot spray coat for 10 mils of a vinyl mastic. This is 10¢ application cost per square foot for 5 mils thickness and 9¢ for 10 mils. Estimating 1 percent replacement of coating after erection (a very high estimate) total surface preparation, material and application cost of a 5-mil protective coating will average just over 25¢ per square foot of steel or about $63.13 per ton.

TABLE 2—Comparison of Methods 1 and 2

	Method 1	Method 2
Initial Capital Outlay per Ton of Steel	37.18	56.63*
Maintenance expense total in 10 years (2) + (3) + (4) + (5) + (6) + (7) of Table 1	286.51	
(2) + (3) + (4) + (5) of Table 2		20.64
Capital write off expense	—18.59	—28.32
Total Capital and Maintenance 10 years	327.40	82.93
6% interest on borrowed capital	22.30	33.98
Total cost per square foot per year	0.131	0.0332
Corrosion Protection Total cost per 1000 Tons in ten years	$327,400.00	$82,930.00
Difference in total cost in ten years, Method 1 and 2		$244,470.00
Tax @ 52% on difference between Methods 1 and 2		$127,124.40
Saving in 10 years per 1000 tons of steel using Method 2		$117,345.60

* Includes credit for lack of shop coat.

Life Should Exceed 5 Years

It is conservative to estimate on the basis of experience, that such a coating will have an almost maintenance-free life of five to eight years, or average six and a fraction years. On a ten year basis, if a single color coat were applied to the entire surface, and 5 percent of the entire surface were spot cleaned and recoated to the original system at the end of six years, the surface cleaning for the color coat, the coat and the application would cost about $22.50 per ton and the spotting as a percentage of the total about $3.16 per ton to maintain appearance and protection, or a maintenance expenditure of about $25.66 per ton.

Rating this expense in terms of 6-year life, the amount spent for the original coating for four years of the second six year period, including the spot recoat, brings the total cost to $81.08 per ton. Adding to this amount the 15% for plant painters' cost above contract painting and subtracting half the original capital cost and the rebate from nonshopcoating, plus capital interest, cost of corrosion protection at the end of ten years of $82.93 per ton or about 3¢ per square foot per year. The steel is fully protected and has been since it was erected.

Coatings considered are vinyls. Other coatings, such as epoxies and phenolics may require additional work for recoating because they are more difficult to apply with good adhesion on recoating. So, costs using these coatings may be somewhat higher than those shown.

Weigh Expense Details

Consider the details of expense with the higher capital plan:

1. Special attention must be paid by the coating workers not to mar or deface erection markings on steel. Marking may be by die or metal tag.

2. Unloading steel to blasting-painting site, turning it, reloading it for erection will take time and effort.

3. The detour through blast-paint operation must be scheduled as tightly as erection. However in modern practice no delays should be expected from weather which are not concurrent with delays in structural steel erection.

4. Special care in erection is necessary to avoid damage to coating.

5. A site for blast-painting operation, where blasting dust will not interfere with erection or operation, must be located where facilities for car loading and unloading of steel and unloading of blasting sand are available.

Summary

By accepting the additional capital cost of approximately $19.45 per ton of steel and assigning to maintenance expense $26.30 per ton over ten years, an estimated expenditure from maintenance funds in ten years of about $263.03 per ton can be prevented.

Further, as a result of the higher capital expense, structure remains in uncorroded condition and excellent appearance after ten years, whereas the structure with the higher maintenance expense has suffered considerable corrosion and is constantly being recoated in areas where the mill scale continues to break loose and tear away the protective coating.

Maintenance and capital costs can be reduced by preferred method from about 13¢ per square foot per year to around 3¢ per square foot per year.

In terms of the average new plant whose coatable steel approximates 4000 tons, the higher capital, lower maintenance method of protection would appear to pay reasonable dividends, even after 52 percent for taxes on the unexpended balance has been deducted.

TECHNICAL REPORTS on MARINE COATINGS

T-1M Suggested Coating Specifications for Hot Application of Coal Tar Enamel for Marine Environment. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-8. Per Copy $.50.

T-1M Suggested Painting Specifications for Marine Coatings. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-7. Per Copy $.50.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

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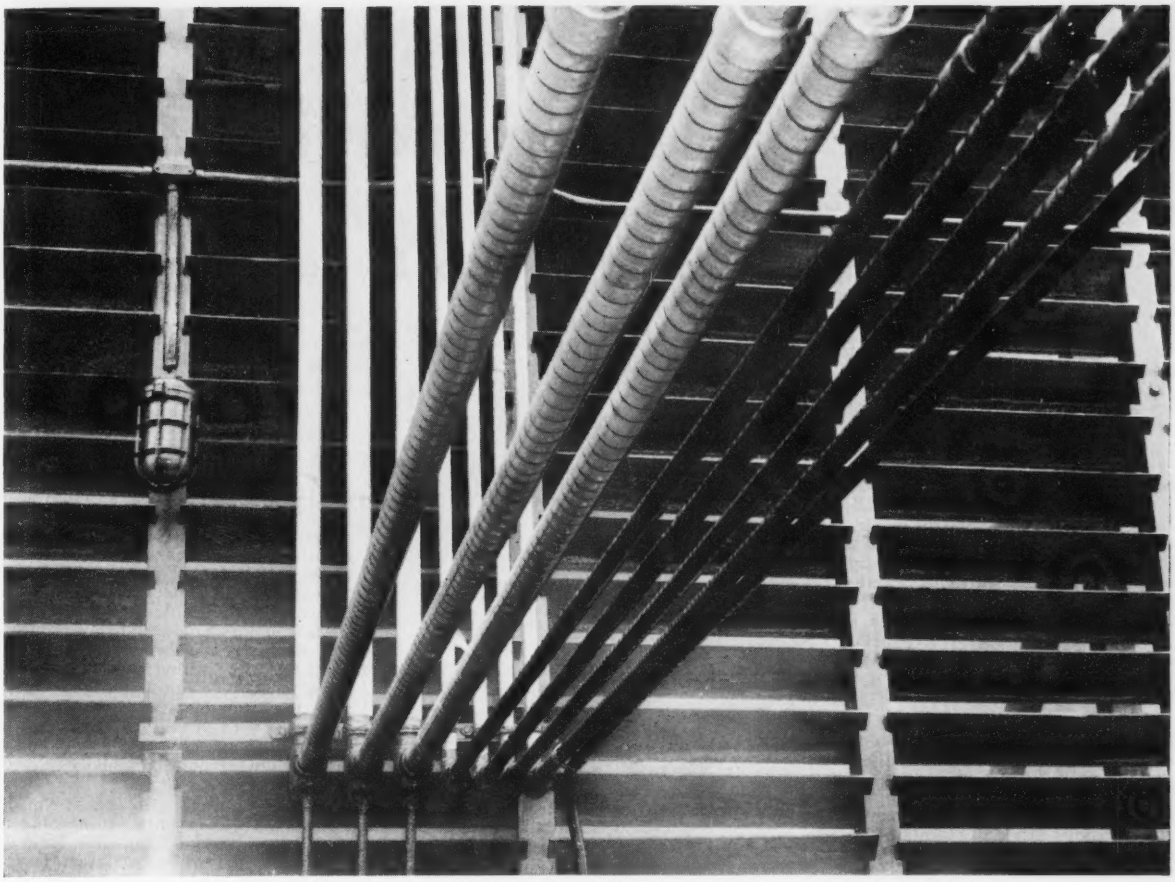

Polyken® Protective Coating keeps this conduit safe from corrosive attack of chemically treated water

Rugged* Polyken *Tape has prevented corrosion damage here since 1951

The conduit pictured above is installed on a water cooling tower serving one of the world's largest fluid catalytic cracking units at Gulf Oil's Port Arthur, Texas Refinery.

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A Review of

Telephone Cable Corrosion Problems*

With Some Suggestions for Improved Methods In Making Surveys of Bare and Ducted Cables

Abstract

Usual practice in laying bare telephone cable in soil and running cables in ducts is described. Common types of cable employed are listed and described briefly. Location of and reasons for location of test points on various kinds of cables and installations are described. Adoption of the copper sulfate/copper reference electrode in preference to the lead slug electrode by many telephone companies is ascribed to changed conditions on underground lead cable. Difficulties associated with measurement of small currents are described as are several methods of measuring potentials of cables underground. Economic significance of corrosion damage on long lines cables is increasing because of the numerous circuits involved in an outage. 7.7

UNDERGROUND plant in the telephone industry can be divided into two categories:

1. Cable plant buried directly in the soil and,
2. Plant placed in conduits, usually of a vitreous clay material.

In addition to these are underground cable dips normally used as an extension of aerial cable plant where continued aerial construction is not practicable.

Corrosion of underground telephone plant similarly falls into two categories; corrosion on "buried" cable plant and corrosion on "underground" cable plant. These two types of plant normally are connected at the outskirts of larger cities so electrical continuity usually exists. In the city proper, cable normally is placed in multiple conduits. This expedites placing, removing and simplifies operations where repeated excavation is not feasible. Economics generally dictate the use of conduit in cities. Similarly, in sparsely inhabited areas or between cities, economics dictate the use of "buried" cable.

Buried and underground cables normally are jacketed in lead alloys. Although polyethelene sheathings have been developed, for the most part lead alloy sheathings are predominant. Lead sheath alloys originally contained 3 percent tin, while present cables contain 1 percent antimony. Lead alloys have an ease of extrudability and flexibility which makes them desirable where sheaths are subjected to a wide range of temperatures and considerable handling.

Buried Plant Is Increasing

Recently buried intercity toll plant has been increasing. This cable is largely lead sheath with protective covering although some has a polyethylene jacket instead of lead. This expansion is due to the fact that existing wire lines can no longer fill circuit requirements and because buried plant enjoys a considerable degree of protection from the elements and thereby a minimum of service interruptions. Lead alloy sheath on buried cables also provides an added shielding from power line induction which is important in many locations.

For testing purposes buried cables can be divided into four categories:

1. Plain lead with jute or thermoplastic protection.
2. Tape armored, gopher protected, wire armored and modified tape armored cables.
3. Thermoplastic copper protected cables (bonded type).
4. Thermoplastic copper protected cable (non-bonded type).

Plain lead sheath cable with a jute or thermoplastic covering involves only a single metal from a corrosion standpoint and corrosion testing on these types involves no unusual features. However, where shield wires for lightning protection purposes are buried along the cable route, care must be exercised to insure that the shield wires do not cause erroneous measurements.

Tape armored, gopher protected and wire armored cables involve a combination of two metals, lead and steel, bonded together with a fairly good conductivity medium between the steel and the lead. Steel is the metal in these types which is closest to direct contact with the soil. Since these two metals are in close proximity to each other in the electromotive force series of metals, corrosion of the steel wires or tapes should not necessarily result in serious corrosion of the lead sheath, as long as no holidays exist in the protective coating between the lead sheath and steel tapes.

With thermoplastic copper protected cable of the bonded type a combination of two metals, lead and copper, widely separated in the electromotive force series, is involved. Metals in this type of construction are connected at each splice point, at approximately 1400-foot intervals. Thermoplastic copper protected cable, in which the copper jacket is kept isolated from the sheath for several miles or more, presents a more complicated problem from a testing standpoint. Two metals are involved, copper and lead, but under normal conditions the lead is kept well insulated from the copper so the problem is primarily that of maintaining satisfactory insulation resistance through the thermoplastic compound. This may be checked by periodic tests of the resistance between copper jacket and sheath.

Buried lead cables must be provided with test points for corrosion testing. These expedite testing and insure more accurate results on corrosion surveys. All buried cables should be engineered to provide ample test points of three general types:

1. Potential test points.
2. Current test points.
3. Cable or foreign structure test points.

Any or all of these types may be involved at a single location, although usually only two are present. Frequency of the test points varies and is determined by various conditions as well as by the type of installation. Potential test points are designed for measurement of potential between the cable and earth. Current test points are designed for measuring the current flow on the cable by means of the millivolt drop over a known length of cable (usually 30 feet). Cable to foreign structure test points are designed to permit measurement of the potential between the cable and a near foreign structure, usually a pipe line.

Potentials of cable to earth can be checked readily on buried toll cable at every gas valve and contactor location where they make direct contact to the sheath at 3000-foot intervals. In addition to standard gas valve locations, potential tests on buried cables can be made at special current test points and at test points normally established at crossings of foreign structures. Standard potential test points normally contain a buried lead slug located four feet from the cable and which is considered a permanent ground electrode. With the introduction of the half-cell, the use of the ground electrode has fallen somewhat into disuse.

In copper jacketed cables where the copper jacket is isolated from the sheath, a single test lead also is connected to the jacket at each valve point. This permits potential and resistance measurements to be made readily between it and the cable sheath.

Current test points locations on buried cable routes are determined primarily by the type of cable and proximity of foreign structures. On almost all types of buried cables, current test points generally are established at intervals of five miles and on each side of and immediately adjacent to repeater stations. In addition to these regular current test points, special current test points are located in areas where foreign structures cross or lie in close proximity. These foreign structures may be pipes or cables underground or a railway operated by direct current or using direct current for signal circuits. Current test points usually are located at such crossings, one immediately at the crossing and one at each side.

In areas where buried telephone cables are parallel to railways or pipe lines and

(Continued on Page 136)

★A paper presented under the title "Telephone Cable Corrosion Problems," by W. W. Elley, Southwestern Bell Telephone Co., San Antonio, Texas at a meeting of South Central Region, National Association of Corrosion Engineers, Houston, Texas, October 18-21, 1955.

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Telephone Cable–

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are within 1000 feet to a mile of such neighboring structures, test points are provided at one mile intervals. If such foreign structures are within 1000 feet of buried cable, current test points usually are located at every splicing pit or at intervals of roughly 1400 feet. A current test point at other than foreign structure locations contain two conductors located 30 feet apart and brought out to a terminal. This provides easy access and permits the measurement of a voltage drop over a known length of cable. At foreign pipe crossings two such locations are provided to permit detection of current flow direction. Cable to foreign structure test leads normally are provided at all pipe crossings using cathodic protection.

Underground Cables Are Bare Lead

Underground cables are confined to relatively small areas largely within metropolitan areas. These are still mostly bare lead in conduit runs with manhole access at a spacing averaging approximately 500 feet. Due to earth stresses conduit joints readily give, causing a crack or small opening which permits silt and soil waters to flow into the conduit. Cement-abestos pipe is gaining favor rapidly as conduit material for underground cables. It is not susceptible to leaks at joints and if properly installed can be kept dry and free of foreign matter. Corrosion failures in pipe dips and risers have been reduced as a result of using cement-asbestos conduit in place of iron pipe.

Small Potentials Are Measured

Extremely small value of potentials under observation today is a primary reason for refining testing methods. Other important factors which have further complicated testing are:

1. Confinement of underground plant largely to metropolitan areas.
2. The close proximity of adjacent underground structures in metropolitan areas.
3. High resistance contacts resulting from the inability to establish electrodes in direct contact with the earth.
4. Independent use of cathodic protection by other utilities.

The last three listed factors are a direct result of the first; the confinement of the bulk of underground plant to metropolitan areas. In metropolitan areas in those sections of cities where the major portion of the surfaces are covered with concrete, asphalt, or similar hard surface material, particular consideration must be given to the location of the reference electrode. High resistance contacts have been observed which render readings from most currently used meters inaccurate. Three possibilities exist to correct for the introduction of high contact resistance:

1. Because of the resistance introduced by conduit, it is permissible to locate the reference electrode at sufficient distances from the cable where earth contacts are available.
2. Vacuum tube voltmeters with resistances of one or more megohms per volt will correct for high contact resistance. These sensitive and expensive meters require specially trained personnel.
3. Where earth contact cannot be made by a remote electrode or highly sensitive meters are not available an alternate solution is to drill a hole through the pavement sufficient in diameter to accommodate a specially constructed reference electrode. Permanent wells with removable covers are recommended where periodic tests are necessary.

Half Cell Being Accepted

A lead slug was accepted as reference electrode in cable corrosion testing for many years but recently a half cell reference electrode has been accepted in the Bell System. The accepted theory supporting the lead slug electrode was that if a material of exactly the same composition as the cable sheath under test were used and completely isolated from outside influences a zero potential would be observed. A lead cable positive with respect to the static lead reference electrode established the cable as the anode and indicated corrosion.

This method and the theory were sound when appreciable increments of current were present and correspondingly high potentials existed. Today, however, it is recognized that a potential difference exists between the lead electrode and the lead sheath under test. This potential difference, although due to several separate phenomena, may be considered as galvanic difference of potential resulting from disimilarity in surface conditions of the cable sheath and of the test plate.

The cable sheath and the test plate thus constitute the two electrodes of a cell which developes a potential difference that has no relation to the current flowing in the structure under test. If the potential developed between the test electrode and cable under test are similar in magnitude to the potential developed between this same cable and earth, the resultant readings are inaccurate. In nonstray current areas most potentials of cable to earth are not as great as those developed between the cable and the lead reference electrode, so readings taken with them are not reliable indicators of either potential or of current flow.

By placing the lead slug in different places or using a different slug it often is possible to obtain a wide variety of small readings. This effect is also possible due to polarization of the slug which results in a change in potential with time. A half-cell now in use by many telephone companies is the copper-copper sulfate half-cell, a stable and predictable reference electrode. For work on lead cables the lead-lead chloride cell has certain advantages but experience has shown that it does not remain stable over extended periods.

Electrode Location Important

For buried cables with a sound protective coating, the reference electrode should be located preferable immediately over the cable. In metropolitan areas, location immediately over the cable is desired but conditions often prevent it. On concrete roads it often is necessary only to dampen the reference electrode thoroughly to establish necessary contact. On asphalt and macadamized roads this does not usually suffice and as an alternative, the half-cell must be taken to a remote location.

The criterion for protection of lead sheathed cables with respect to a copper-copper sulfate half-cell has not been accurately determined. Based primarily on observation and experience, a value of —.75 appears satisfactory.

Potential-To-Earth Measurements

Over-all studies should involve single cable-to-earth potential measurements at points where relatively easy contact can be made to the cable normally at manholes.

To assist in analysis of cable-to-earth potentials it often is advisable to plot observed potentials-to-earth readings along the cable run. This readily locates positive areas and other significant conditions.

Where conditions exist which require detailed study and extensive tests, use can be made of one or more half-cells in cable-to-earth measurements if one important principle is observed: If two identical half-cells are placed at separate points, the potential difference between them is substantially due to IR drop in the earth. Thus a reading from a cable to a half-cell may not have special significance, but the difference between readings taken between a cable at a fixed point and a half-cell placed successively at various points on the earth will give an indication of the direction of current flow in the earth.

Duct Surveys Are Replaced

This principle is applicable on a line perpendicular to the cable, on a parallel line or on both simultaneously.

A single half-cell in series with a meter through which contact is made to the cable can be used to provide data formerly acquired through duct surveys. In place of the lead slug electrode pushed through the duct, with potentials read at intervals, the half-cell can be moved on a line parallel to the cable and contact made periodically to the earth on the outside of the duct. Potentials are considered to reflect the average potentials of the cable over a length approximately four times the distance of the half-cell from the cable. If the half-cell is located ten feet from the cable, the potential observed will be the average for 40 feet of cable. These values can be plotted so points of higher potential indicate corrosive areas.

A more elaborate and more accurate extension of this test involves measurements of potential between the cables and a half-cell placed successively directly over the cable and at points on either side of the cable. Using the measurement of cable to half-cell directly over the cable as a reference, the direction of the current in the earth may be deduced. Table 1 gives an idea of the magnitude of the potentials that may be encountered, along with the interpretation in terms of current direction.

A series of such tests over a suspected section can pin-point corrosive areas.

IR Drop Measurements

In computing current from potential drop it is necessary to correct the meter reading for resistance of test leads and divide the correct potential by sheath resistance. Sheath resistance is never accurately known, particularly in multiple cable runs, so any attempt to measure IR drop in successive sections is likely to provide dubious results because possible errors are larger than the quantity sought. However, current measurements with instruments now in use show direction of currents from two or more directions into a section of cable.

(Continued on Page 137)

Telephone Cable–

(Continued From Page 136)

When improved instruments and test procedures are developed, measurement of current and current loss will become more important in testing procedures in non-stray areas.

Conclusion

Corrosion of lead sheathed cables is becoming increasingly a major problem in telephone companies. Local action accounts for a continually growing number of failures since abandonment of track return trolleys. Stray current losses are increasing sharply due to installation of cathodic protection on other utilities' underground plant in congested areas. The cost of replacing cables is becoming increasingly high.

The multitude of circuits common to a cable increase revenue lost as a result of a failure. The problem is aggravated because testing techniques are not fully adapted to small currents encountered. These currents may be attributed to the following causes:

1. Differential aeration.
2. Concentration cells.
3. Stray currents from foreign cathodic protection systems.
4. Galvanic currents.
5. Long Lines currents.

Lead, an amphoteric metal, reacts to bases or acids. Extensive use of cathodic protection on cable must be given careful consideration to avoid excessive negative potentials creating base solutions adjacent to sheath in sufficient concentration to result in cathodic corrosion.

A properly engineered corrosion plan must consider not only economy in installation and operation but also a joint protective system involving all utilities operating in the area. No more bare lead sheath than absolutely necessary should be laid underground. Cables with a polyethylene sheath placed externally over thin sheaths of lead, aluminum or steel are referred to as polyethylene jacketed, alpeth and stalpeth. They are a practical solution to many of corrosion problems.

Organic Solvents Control Next Air Pollution Aim

About 400 tons of organic solvents are entering the atmosphere of Los Angeles county daily as vapor, according to the county's Air Pollution Control District. These vapors come from such sources as paints, dry cleaning plants, rotogravure plants, paint manufacturers, automobile assembly plants, aircraft companies, manufacturers of cans and containers, furniture and appliances. The amount is estimated to be about one-fifth of the daily organic emissions.

An investigation is underway now into the feasibility of legislation limiting emission of these vapors into the air. A measuring device using infra-red radiation to measure carbon atoms is under consideration. It would be used to scan air entering and leaving a plant where solvents are used and compare the number carbon atoms.

When a means of measuring solvent emission has been developed legislation leading to control of industries involved will be prepared. Controls will be sought for segments of industry as rapidly as efficient measuring devices can be contrived.

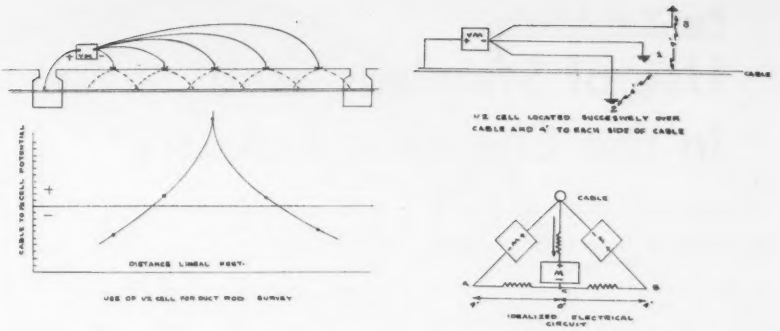
CABLE TO VE CELL POTENTIAL
DISTANCE LINEAL FEET
USE OF VE CELL FOR DUCT ROD SURVEY
VE CELL LOCATED SUCCESSIVELY OVER CABLE AND 'A' TO EACH SIDE OF CABLE
CABLE
IDEALIZED ELECTRICAL CIRCUIT

Figure 1

CABLE TO EARTH POTENTIALS			Interpretation of Current Direction
Point C	Point A	Point B	
+0.2	+0.25	+0.26	Leaving
+0.2	+0.10	+0.11	Leaving
−0.05	0.0	+0.01	Leaving
+0.01	+0.15	−0.10	Transverse

The purpose of making the measurements is to determine the potential of C with respect to point A and B and thus to deduce the direction of current flow. The potential of C with respect to A is equal to the potential of the cable with respect to A less the potential of the cable with respect to C. Similarly the potential of C to B can be found. It is important to note the values are the algebraic summations and due regard must be given the sign.

EXAMPLE

A = + .25 C = + .20	B = + .26 C = + .20
Cable = + .05	Cable = + .06 — Current Leaving
A = + .10 C = + .20	B = + .11 C = + .20
Cable = — .10	Cable = — .09 — Current Entering
A = 0.00 C = — .05	B = + .01 C = — .05
Cable = + .05	Cable = + .06 — Current Leaving
A = + .15 C = + .01	B = — .10 C = + .01
Cable = + .14	Cable = — .11 — Current Transversing

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Part 2 of 2 Parts

Use of Stainless Steel to Combat Corrosion In the Chemical Industry*

(Continued from Sept. 1957 Issue)

Characteristic Defects and Remedial Measures

It is important to recognize the limitations of the stainless steels as regards both inherent weaknesses and those which may arise from design. Four major characteristic defects are susceptibility to pitting and concentration cell effects, sensitization, stress corrosion cracking and hot shortness.

A. **Pitting and Concentraion Cell Attack.** It is difficult to differentiate between these phenomena. Nuclei for pitting probably are engendered most often by action of small penetrating chloride ions on the otherwise protective surface film. Subsequently, there may come into play oxygen concentration cell effects, ionic concentration cell effects and galvanic relationships between the active anodic area and the large passive cathode area surrounding it.

Stainless steel frequently pits under deposits in cooling waters or in process environments and are common under rust deposits in stainless lines handling soft waters in which there has been previous corrosion of galvanized, steel or cast iron equipment. For some reason, the insoluble iron carried in the corrosive water tends to precipitate at places where there has been a disturbance in flow, such as around welds or other protuberances in the stainless equipment. Even in steam condensate, for which stainless steel normally is considered the complete solution as far as corrosion is concerned, there is a tendency to pitting if rust deposition occurs.

Probably the most common occurrence of concentration cell pitting of stainless is on gasket faces, as described by Kunkel.[16] He cites the adverse effect of using long fiber asbestos gaskets, which can have a wicking action in the flanged area with attendant oxygen concentration cell pitting. He says rubber faced gaskets and gasket materials without asbestos did not seem to cause serious pitting. However, the use of short fiber asbestos is not always practicable. The writer has encountered pitting on stainless gasket surfaces apparently caused by this type of action. However, the equipment in question was in high pressure service and a recommendation that short fiber asbestos be used brought out the fact that short fiber material does not have the physical strength for high pressure application. The decision in such a case must be a compromise.

In general, freedom from pitting of stainless steels can only be assured in environments in which there is no tendency to form localized deposits, and even then it is difficult to maintain pit-free surfaces in gasket areas.

B. **Sensitization.** Sensitization, or "weld decay," of the austenitic stainless steels is probably their best known deficiency. This phenomenon is very striking, is readily reproduced and can be studied readily by metallographic and corrosion investigation. For these reasons, sensitization has been widely publicized. Unfortunately, the number of references in the literature on the topic frequently lead to measures against this type of attack in environments which are, in effect, innocuous even to critically sensitized material. It should be emphasized that intergranular corrosion of sensitized stainless steel is contingent on the corrosive environment.

The most commonly accepted theory explaining sensitization attack is that when austenitic stainless steels are held in the temperature range 800 to 1500 F, carbon migrates to the grain boundaries and precipitates as chromium carbides. This leaves a chromium-deficient area around each grain, which is subject to preferential corrosion in certain environments. This explanation appears to be adequate for the 18-8 stainless steels, 304, 321, 347, etc. It does not satisfactorily explain the behavior of molybdenum-bearing grades.

Apparently, another metallurgical constituent, "sigma phase," enters into this mechanism[9] as indicated under the section "Specifications." An allied form of attack is "knife-line" attack, so designated by Holzworth.[17] In the area immediately adjacent to the weld of a stabilized material, the titanium or columbium carbides may be redissolved by the high heat of welding. This effectively takes the stabilizing elements out of play, so that *subsequent* heat treatment in the sensitizing range renders them susceptible to corrosion in a very narrow line on each side of the weldment. It is said that Type 321 is more susceptible than 347 to this knife-line attack.

Two major methods of avoiding sensitization are to use stabilized grades, which contain titanium, columbium, or columbium-tantalum, or the extra low carbon grades. Occasionally a type of stabilization heat treatment at 1600 F designed to precipitate *spheroidized* carbide particles to the extent they do not promote intergranular corrosion is used. This heat treatment generally is not considered a practical solution.

The use of the low carbon grades of stainless steel has been the subject of considerable study.[18, 19] Early investigations by Bain, Rutherford and Aborn had indicated that the susceptibility to intergranular corrosion should cease at around .03% carbon. Heger and Hamilton's linear regression analyses of these data, however, indicate that a carbon content of less than .009% is necessary for complete freedom from intergranular attack.[19]

It would appear that many investigations tend to be academic in that they are pointed toward *absolute* freedom from intergranular corrosion under conditions of long exposure in the sensitizing range and simultaneous or subsequent exposure to corrosive agents specific for intergranular attack. These conditions normally are not encountered in chemical process corrosion. In practice, the writer feels that the conventional 0.03% maximum 18-8 stainless steels are adequate for most chemical plant processes.

An interesting question arises from the fact that low carbon grades of Type 316 stainless steel cannot be tested by the Huey Test. The question naturally arises: Does the failure of the molybdenum-bearing grades to perform as expected in the boiling 65% nitric acid test reflect upon the test or upon the material? For example, it has been argued that this indicated that the test was no good. It also has been argued that this means that the low carbon grades of 316 will not perform in service in a manner analogous to low carbon 304. The writer believes the answer hinges entirely upon the corrosive environments to which they are exposed. In field corrosion tests in the process streams at the writer's Texas City plant, low carbon grades of 316 generally perform satisfactorily when sensitized, even in environments in which sensitized regular carbon content 316 is rapidly corroded. This experience indicates the use of low carbon 316 is feasible. On the other hand, it should be borne in mind that if there is one environment (e.g. boiling 65% nitric acid) which attacks sensitized low carbon 316 in an intergranular manner, then there well may be other process liquors which will do the same thing. At present it is impossible to predict this occurrence by a chemical analysis of the stream, so it becomes necessary to make extensive laboratory or field corrosion tests before electing to use a low carbon 316 in an unknown process. The same is true of columbium-stabilized 316, which likewise tends to fail the Huey Test.

C. **Stress Corrosion Cracking.** This apparently is the most serious defect of austenitic stainless steels used in the chemical process industry. Susceptibility of the austenitic stainless steels to this type of attack has been overshadowed to a great extent by the volume of literature on sensitization. The writer has seen many failures in which stress cracking was responsible, and very few in which sensitization was a factor. Incidence of stress corrosion cracking probably have been obscured largely by the fact that it has not been widely reported among the myriad services in which stainless steels are employed which include not only the chemical process industry but such things as architectural trim and service for anti-contamination. In many slightly corrosive environments, and particularly where brackish cooling water is employed, this is a major problem. Stress-corrosion cracking has been reported in such diverse media as water, condensate,

(Continued on Page 140)

★ A paper by Charles P. Dillon, Union Carbide Chemicals Corp., Texas City, Texas presented at a meeting of Niagara Section, National Association of Corrosion Engineers, at Buffalo, N. Y., May 9-10, 1956.

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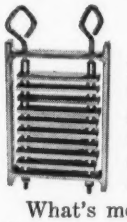

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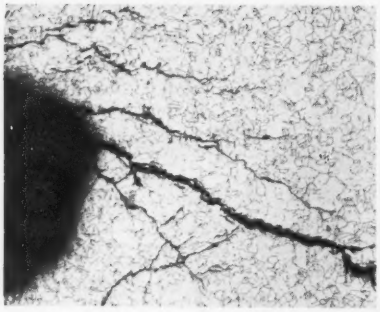

Figure 5—Photomicrograph of stress-corrosion cracking in thread root of Type 304 bolt. Medium: Wet salt-contaminated insulation.

Figure 6—Stress-corrosion cracking of Type 347 pipe in a process effluent.

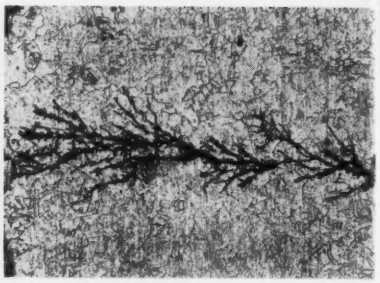

Figure 7—Photomicrograph of stress-corrosion cracking of Type 347 pipe.

Use of Stainless—

(Continued From Page 138)

acetic acid, caustic, coffee, baked beans and tomato soup.

Some idea of the frequency of failures can be gathered from the following examples:

Figure 5 shows a Type 304 stainless steel bolt which failed in four years at 80 C, due to salt air and moisture contamination (from the atmosphere) of hair felt and magnesia insulation.

Figure 6 shows a section of Type 347 stainless steel pipe which failed by cracking in a process effluent. Typical branching transcrystalline penetration is shown in Figure 7.

Figure 8 illustrates the stress corrosion cracking by brackish cooling water of two 18-8 condenser tubes. Observe that one has cracked due to residual hoop and tensile stresses arising from the forming of the tube and the other, from a hairpin bundle, shows distinctly the effect of the tensile stresses imposed by the bending of the tube.

Type 347 autoclave brine jackets have been observed to crack in 20 months' time by the action of chromate-inhibited 28% calcium chloride brine at a pH of 8.5, when temperatures ranged from —10 C during normal service to 190 C during steaming-out operations. The stresses arose from differential expansion between the solid, light gauge brine jacket and the heavier stainless-clad steel wall of the autoclave proper.

Figure 9 shows the cracking of a Type 304 preheater in process water contaminated with chlorides and with a small amount of acetic acid. The stripping still which operated with this unit also failed in 18 months. Construction was of mixed Type 316 and 347, operating temperature was 128 C, pH was 4 to 6 and there was about 250 ppm of chlorides in the environment.

Type 304 stainless steel preheaters cracked in 20 months in an environment analyzing 99.9% water, 100-300 ppm acetic acid and 30-40 ppm chloride at a pH of 3-6. The temperature was 130 C. The failure is illustrated strikingly by the cracking of the tube-sheets shown in Figure 10. This water had been made oxygen-free by addition of sodium sulfite. It is not certain what the cathodic depolarization reaction was in this instance, although it has been established definitely that stress-corrosion cracking is an electrochemical phenomenon.

Among other instances is the failure of Type 316 heating coil tubes in a batch still for chlorinated hydrocarbon

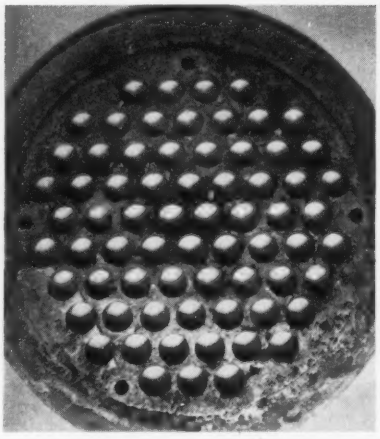

Figure 10—Stress-corrosion cracking of Type 304 tube-sheet in sodium sulfite-deaerated process water contaminated with acetic acid and chlorides.

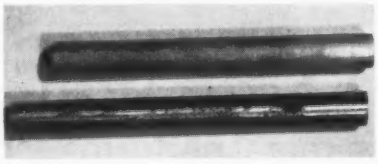

Figure 11—Stress-corrosion cracking of Type 316, pitting of Type 329 condenser tubes by recirculated cooling water in vertical condenser.

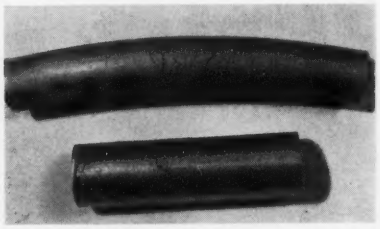

Figure 8—Stress-corrosion cracking of Type 304 condenser tubes in recirculated cooling water by residual and by applied stress.

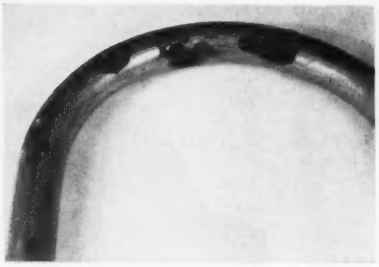

Figure 9—Stress-corrosion cracking of Type 304 condenser tubes in process water contaminated with acetic acid and chlorides.

service in 10 months at 125 C kettle temperature. Of course, the wall temperature of the tubes was considerably higher. In another instance, a 316 calandria failed in four years in a solution of 18% sodium chloride containing 0.2 to 0.3% sodium hydroxide at 135 C. In still another case, a 316 condenser failed in eight months in cycle water due to residual stresses. The process side was 132 C. Recently, due to "minor operating changes," a 316 heating coil failed by cracking *in three weeks* where the tubes were expanded into the tube-sheet. Previous coils had a life of up to 5 months.

Figure 11 shows a Type 329 and a Type 316 tube which have been in service side by side in a vertical stainless condenser. This condenser has a process inlet temperature of about 150 C, with water countercurrent on the shellside having a discharge temperature of 60 C. The 316 tube failed by stress-corrosion cracking in the first 6 to 10 inches below the upper tube sheet in 18 months. Attempts to use a duplex alloy as a preventive measure resulted in severe pitting under the same conditions, although this may have been engendered by faulty heat treatment of the Type 329.

Most investigators consider Types 304 and 316 stainless steels approximately equal in their susceptibility to stress corrosion cracking.[20] Other investigators seem to feel that Type 316 may be superior,[21] a feeling probably due to the superior resistance of 316 to chloride pitting. It is alleged, quite reasonably, that cracking must begin as pitting, at least on a microscopic scale. It is sometimes felt that the extra highly alloyed stainless steels are immune to cracking, but cracking of a 20 chrome-20 nickel alloy has been reported.[21] Zappfe[22] reports that German metallurgists believe that the higher austenitic alloys, which are more stable than 18-8, are less susceptible to stress corrosion cracking.

An important consideration in any discussion of stress corrosion cracking is the applicability of alleviating stress-relieving heat treatment. Zappfe[23] refers to a stress-relieving heat treatment on the order of 800 F, and mention is made

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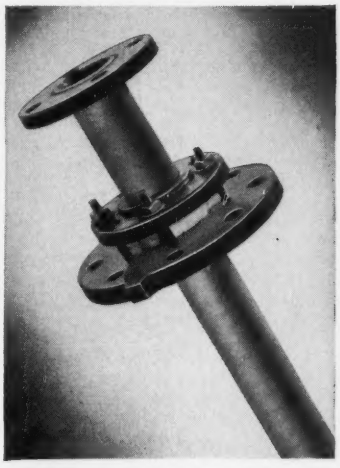

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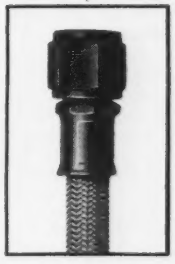

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Use of Stainless—

(Continued From Page 140)

frequently of this treatment in the handbooks. It should be noted that this is designed to improve mechanical properties after cold-work and has little applicability to the phenomenon of stress-corrosion cracking. Thielsch,[24] for instance, notes that stress-relief is less than 50% effective when conducted at 1100 to 1250 F and is 90% effective at 1600 F.

Experiments on low temperature stress-relief were conducted in the writer's laboratories on miniature tensile specimens stretched against a supporting frame. Stress was calculated from the strain applied. Beam-loaded specimens, using Baldwin strain gauges, were employed also. For specimens prestressed to 65,000 psi and subsequently stressed to 50,000 psi, it was found that a stress-relieving heat treatment at 800 F achieved a maximum of 50% relief and sometimes as little as 20-25% relief for heat treatments on the order of several hundred hours per inch of thickness. On the other hand, heat treatment at 1500 F for 164 hr./in. thickness gave 99% stress-relief. For practical purposes, the writer prefers a stress-relieving heat treatment at 1650-1750 F at two hours per inch of thickness.

It is noteworthy that when a given environment has been established as specific for stress-corrosion cracking but does not cause accelerated attack on sensitized stainless steel, the writer has no hesitation in applying a stress-relieving heat treatment (furnace-cooled) to unstabilized or regular carbon grades of stainless steel. If, on the other hand, the environment also will cause intergranular corrosion, then the use of stabilized or extra-low-carbon grades is indicated. These can be stress-relieved without detriment to their corrosion resistance.

Behavior of the straight chromium alloys, the chromium-nickel-manganese alloys and the duplex alloys is unpredictable in environments conducive to stress-corrosion cracking. It seems that all can be susceptible to stress-corrosion cracking, depending on the environment. The first two groups, particularly, have been reported as failing in dilute salt solutions or in salt spray. Committee T-5E of the NACE has been formed on Stress-Corrosion Cracking, and any data that may contribute to a knowledge of the basic cause of this sort of failure, or practical palliative measures, should be reported to it. The possible beneficial effects of couples between stainless tubes and copper or steel shells has been discussed earlier herein. Conversely, stress-cracking of stainless steel can be hastened by making it the anode in a galvanic couple, although such couples probably will not be encountered often in the chemical industry.

It sometimes is asked whether cast materials can be subject to stress corrosion cracking. There seems to be no reason why a cast stainless steel should not be subject to this type of failure except that it is less likely to contain residual stresses from fabrication than corresponding wrought equipment. An instance has been reported in which a cast ACI-CH (25-12) pump impeller had failed by cracking in magnesium chloride brine. This was due apparently to operational stresses imposed on the impeller. In this case, the failures were successfully prevented by giving the impeller a stress-relieving heat treatment in the sensitizing, or carbide precipitation, range. This would not affect operational stresses, but apparently lowered the overall corrosion resistance slightly to a point at which stress corrosion cracking could not set in. Stress corrosion cracking is contingent on precisely defined conditions of stress and corrosion and does not occur usually in either wholly non-corrosive or in moderately corrosive environments.

This approach is not generally applicable. It is necessary to determine whether or not stress corrosion cracking and sensitization may not be simultaneous factors in a given process. In the writer's experience, usually they are not, with stress corrosion cracking more common.

When stress corrosion cracking occurs in annealed stainless it is typically transcrystalline and multi-branched. (A single transcrystalline crack usually denotes fatigue.) If the metal has been sensitized, it may fail in either a transcrystalline or intergranular manner, depending upon the environment. While it was thought originally that all stress-cracking of stainless was intergranular in nature, this proved later to its identification mistakenly with sensitization effects. Most failures are typically multi-branched transcrystalline cracks.

D. **Hot Shortness.** In fabrication by welding, the hot short cracking of stainless steel is observed primarily in the columbium stabilized grades and to a lesser extent in the molybdenum-bearing steels. It is due apparently to a low melting eutectoid formation at the grain boundaries at elevated temperatures, such as are encountered in welding, which results in diminished strength such that any applied restraint or stress results in microfissures at the grain boundaries. These may propagate to macro-cracks in some indeterminate period ranging from several hours to several months or even years. Wholly austenitic alloys are more prone to this type of attack than those which contain small amounts of ferrite, hence the 309 and 310 grades have a tendency similar to 347 in this regard. (However, the reverse is true in hot-forging operations.) There are numerous references in the literature as to practical methods of alleviating this defect.[24, 25, 26]

Fabrication

Reference has been made to the relatively poor weldability of the straight chromium grades of stainless steel and to the excellent characteristics of the austenitic grades in this respect. Certain considerations, however, should be constantly in mind with regard to the welding of the latter grades.

The general rule in the selection of stainless steel weld rod is that it should be of essentially the same composition as the base metal. Frequently the rod is more highly alloyed in order to make up for slight losses in outgoing elements during the welding process. For instance, on Type 304 it is common to use 308 (19-9) electrodes. Further, in the welding of the Type 321, a 347 rod is preferred because it will transfer columbium across the arc whereas there is a tendency to lose titanium across the arc if a 321 rod is used. Type 310, a 25-20 alloy, is used normally for joining stainless to steel because the use of lower alloy rods would result in a weld ment of a lower alloy content having definite air-hardening characteristics, leading to loss of ductility.

Because of the hot short characteristics of 347 previously noted, difficulty is sometimes encountered. In heavy 347 forgings eutectic columbium compounds are believed to initiate base metal cracking during welding.[27] A 347 rod with a lime type coating and about 5% ferrite is a definite help in this regard.[28] Several applications have been noted in which hot short cracking of 347 occurred and it was determined that it was a misapplication and that 347 was not needed for corrosion resistance. In such cases hot short cracking has been combated by using Type 309 (25-12) rod. It has been observed that crater cracks at the ends of welds, or possibly a slight difference in corrosion resistance of weld metal in the crater, occasionally can start severe corrosion.[26]

Thielsch[24] has published a very comprehensive paper on the welding of stainless steels.

An excellent summary of the principles of design, fabrication and installation to acquire optimum performance of stainless steel equipment was published by Collins.[9] In addition to the standard considerations with regard to notches and crevices, it is important not to have excessive cold work in vessels of welded construction. Excessive cold work may result in cracking during the welding process. It has been suggested that the tendency to cracks and warpage on welding is a function of the superficial hardness. Superficial hardness tests indicating values greater than 70 on the 30 T scale are considered to be excessive and may lead to welding difficulties, whereas superficial hardnesses of 65 or less on the same scale are considered acceptable. For pressure vessels, certain limitations are sometimes put upon the degree of cold working allowed before annealing should be specified.

Fabrication Method Important

The mode of fabrication apparently has some effect upon the extent to which the metal will be sensitized. For instance, it shoud not be assumed, because one has welded stainless steel, that sensitization has in fact taken place alongside the weld. As indicated by a survey by the Chemical Industry Advisory Board of the American Standards Association, the extent to which stainless steel is sensitized on welding is dependent upon the thickness of the metal and the welding technique used. It has been observed that sensitization often is worse in the thinnest sections, although the contrary has been reported occasionally.

It might be expected from the concept of severity of quench that very thin sections are less adapted to remove heat from the heat-affected zone, whereas in plates of greater thickness heat is more readily conducted away so that one approaches the severity of quench of a typical water-cooling effect. It has been reported in the literature that sensitization of weldments can be minimized by water-quenching the welds as they are made, provided rapid cooling is effected within two minutes from the time of deposit of the bead. At the writer's laboratory an attempt was made to determine whether or not such quenching had any effect during the weld-

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TYPICAL CORROSION RESISTANCES OF ZICRONIUM AND TITANIUM

CORROSIVE MEDIA	METAL RESISTANCE	
	ZIRCONIUM	TITANIUM
Sulfuric Acid	excellent to good below 80%	good below 5%
Nitric Acid	excellent	excellent
Hydrochloric Acid	excellent	good below 10%
Phosphoric Acid	excellent to fair below 85%	poor
Chromic Acid	excellent	excellent to good
Aqua Regia	poor	excellent
Wet Chlorine Gas	poor	excellent
Chlorine Water	excellent	excellent
Sodium Hydroxide	good below 90%	good below 50%
Ferric Chloride	poor	excellent
Calcium Chloride	excellent	excellent
Cupric Chloride	poor	excellent
Sodium Chloride	excellent	excellent
Ammonium Chloride	excellent	excellent
Aluminum Chloride	excellent	excellent to fair

Corrosion resistances of zirconium and titanium to typical chemicals are shown above.

At right is a steam jet made of zirconium which has given trouble-free performance after a year in hydrochloric acid service. For comparison, a throat piece from a steam jet made of cast iron is shown after only a week of similar service.

ZIRCONIUM AND TITANIUM LICK TOUGHEST EQUIPMENT CORROSION PROBLEMS

The use of zirconium, titanium, or alloys of these metals now makes it possible to fabricate equipment that is corrosion-resistant to almost every substance encountered in industry. Even with such difficult-to-handle chemicals as chlorides and oxidizing acids, equipment can have extremely long service life. Problems of product contamination in chemical and food processing can be virtually eliminated.

Costs are cut in two ways. Direct maintenance costs are lowered, but even more important, costly downtime for the replacement of equipment can be avoided. These savings can more than compensate for the relatively high initial investment.

At the moment, equipment can be fabricated from titanium for about twice the price of stainless steel with even lower costs anticipated in the near future. When volume production is reached, commercial grade zirconium will probably cost only 50 to 75% more than stainless steel.

Wider application of these metals at reasonable cost is made possible by increased production and improved techniques provided by new U.S.I. facilities. By the end of this year, U.S.I.'s new titanium plant will add 5,000 tons to the nation's annual output. The new U.S.I. zirconium plant at Ashtabula, Ohio, employing a sodium-reduction process, will be able to supply 1,000,000 pounds of zirconium platelets to industry in 1958, in addition to meeting Atomic Energy Commission commitments.

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Use of Stainless–

(Continued From Page 142)

ing of 316 in thicknesses up to ½-inch. Results showed none of the specimens, whether water-quenched or as-welded to be susceptible to intergranular attack in qualification tests. On the other hand, tests conducted on pipe at the South Charleston laboratory of Union Carbide Chemicals Company definitely indicated that sensitization was occurring on each side of the weld and that water-quenching was not effective. The writer inclines to believe there are no adequate quantitative data on this matter and that there is little likelihood adequate data can be obtained because welding remains an art rather than a science. Again, this problem is perhaps not of the magnitude ascribed to it by many because, if sensitization is known to be a factor in a given process, it is wise to use extra low carbon or columbium stabilized grades of stainless in the first place. There is some reason to believe also that adequate investigation of the likelihood of sensitization in a given process stream probably would effect as great a saving of columbium, titanium and tantalum as could be accomplished by investigating the techniques of welding to minimize weld decay.

Repair of Pump Shafts

An interesting problem arises sometimes in connection with the repair of stainless steel pump shafts. It is quite common, for instance, to find that an alloy stainless steel pump shaft has been deteriorated by corrosion in such a fashion that it must be replaced. So it sometimes is desirable to weld a new stub end to the shaft rather than to replace it all. Difficulties have been encountered, particularly with the high alloyed chrome-nickel-copper-molybdenum metals, in this type of application. Attempts to weld 316 stub ends to these super alloys frequently results in cracking failures in the fusion zone between the weld and the super-alloy stainless. This has been minimized by using a technique of buttering both the stub and the end of the alloy shaft before welding. However, more recently it has been found that the new alloy rod marketed by the International Nickel Company under the name "Inco Rod A" has superior characteristics for the welding of such dissimilar metals. Prior to the use of Inco Rod A, the best technique available consisted of buttering the stub end of the 316 and the higher alloy material and welding with a Type 310 rod. In this case, tensile strengths of the order of 55% of the super alloy material were effected. When similar welds are made between 316 and the high alloy stainless using an Inco Rod A, strengths in the weldment approach 95% of the ultimate strength of the chrome-nickel-copper-molybdenum alloy.

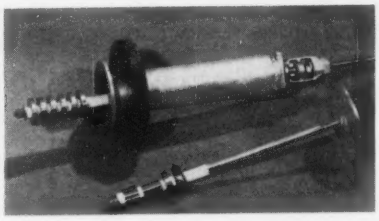

Figure 12—Insert and slip-in field corrosion racks.

Corrosion Testing

A few remarks are in order about techniques of field corrosion testing of stainless steel for the chemical industries. Many companies supply specimens in a standard size which facilitate computation of corrosion rates in terms of milligrams per square decimeter per day. The writer's experience with these specimens is that they often are unhandy if entry is to be made into existing equipment. In cases where the specimens are exposed on spool type racks, this is a small matter because such racks usually are installed and removed only at times of major shutdown. Frequently, however, it is necessary to provide easier means of entry into process equipment.

Specimens on the order of 1¼-inch OD to 1½-inch OD suffice for 95% of the writer's test work. Such specimens made with a 7/16-inch diameter hole can be supported readily on corrosion racks fabricated from ¼-inch rod and covered with ⅜-inch Teflon insulation. They also are small enough for entry into pipe or nozzle apertures as low as 1½-inch tubing. For smaller apertures it sometimes is necessary to prepare corrosion specimens from lengths of condenser tubing.

The conviction is reiterated that welded specimens accomplish very little unless the aim is to learn something about characteristics of a weld deposit on the metal. For purposes of studying heat effects the writer prefers to duplicate the anticipated heat treatment or, even better, to maximize it. For example, in studying the effect of sensitization following procedure is recommended: Specimens of the stainless steels to be tested should be prepared as corrosion coupons and heat treated for two hours at 1250 F. As reported by Binder, Brown and Franks[18] this should effect maximum sensitization. In the event a specimen so treated does not show significantly greater corrosion than that which occurs on a properly annealed specimen, it is concluded that sensitization phenomena will not be encountered in the process. This criterion has been used successfully for many years and has yet to be found unreliable.

It is necessary occasionally to run field corrosion tests on susceptibility to stress corrosion cracking. This may be done with U-bend specimens, beam type specimens or specimens specifically fabricated from crushed condenser tubing. One difficulty with such tests is that hot wall effects which frequently contribute to stress corrosion cracking failures in service cannot be duplicated. Nathorst[21] has published a dissertation on the suitability of the U-bend specimens in such investigations.

Two of the techniques used for field corrosion testing are illustrated in Figure 12. The top rack is designed for insertion in an unused pressure vessel nozzle. It allows fairly ready removal of the rack without actually effecting entry into the vessel proper. On the other hand, it requires that the equipment be out of service if it is inserted or removed. The bottom rack is typical of those used for many investigations in which it is essential that corrosion rates be studied over a definite period of time and when it is necessary to get in and out of the operating equipment without shutting it down. It consists essentially of a flange, a recessed area for the coupons, a packing gland arrangement made from the bonnet of a ½-inch stainless valve and a long rod with the specimens mounted on the end. By mounting a gate valve to a spare nozzle, it is possible to attach this rack to the equipment, or take it off, without taking the equipment out of service. The rack is bolted to the valve, the valve opened and the specimens on the rod slid into the operating equipment for exposure. They can be removed at any time regardless of operating condition. This type of rack is, of course, limited to relatively low pressure services. For high pressure services it is possible to effect similar rigs using a "Scotch Nipple." However, in such an

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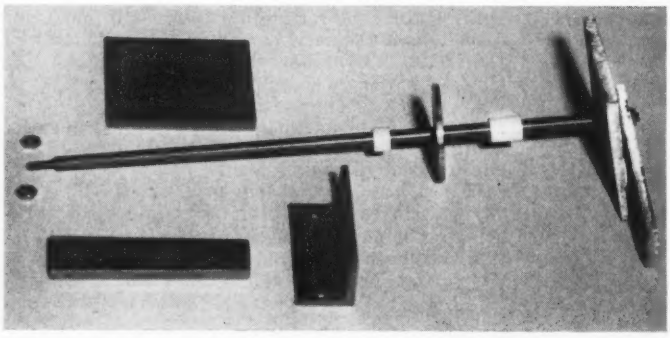

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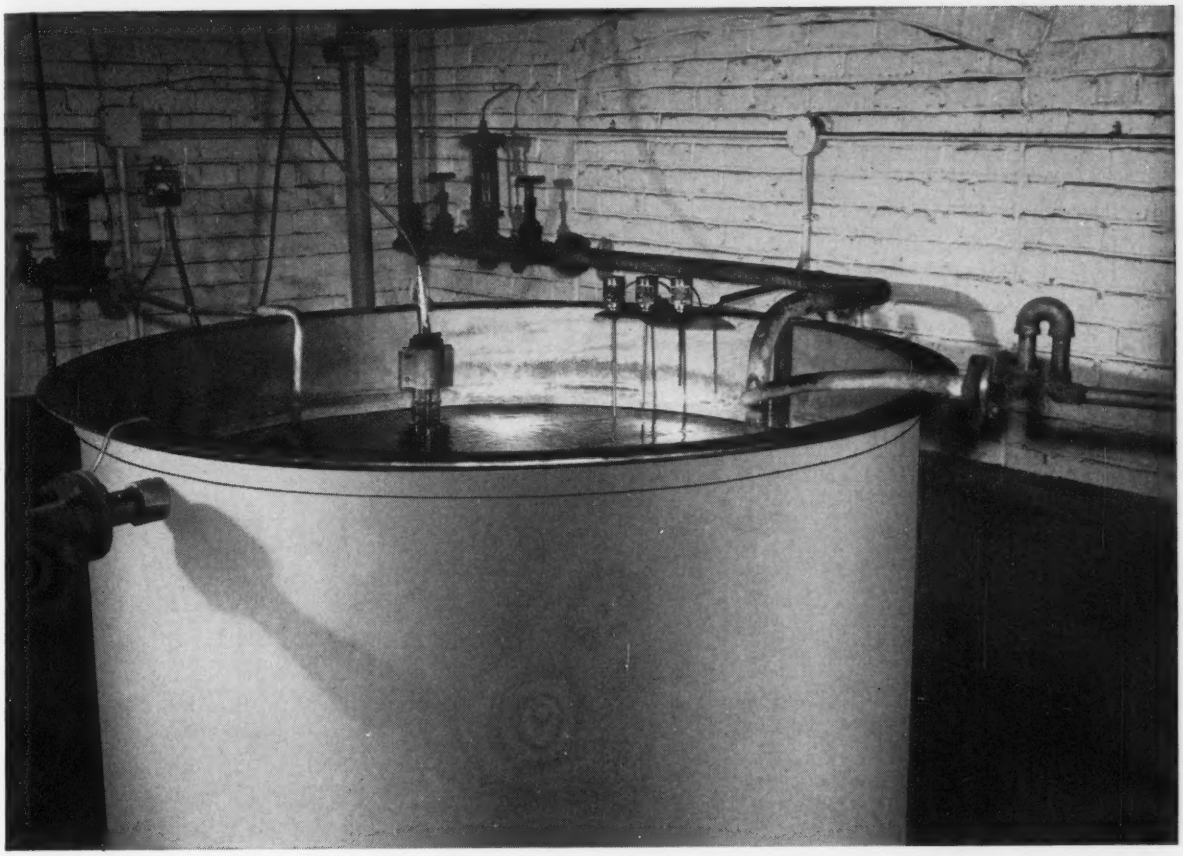

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Use of Stainless–

(Continued From Page 144)

event one is limited as to the number of specimens which may be employed.

Conclusion

Not many years ago stainless steel was considered to be something of a wonder metal. In many applications today the engineer finds himself in a position of wondering what on earth is going to happen to it next. The performance of stainless steel in chemical process service is contingent upon proper selection. Poor performance usually follows improper selection, careless fabrication procedures and/or operational changes without adequate study of their effect.

Specifications are no substitute for common sense. Many times precious alloy components are wasted because the project engineer insists upon going "first class." In other cases, intolerable delays are brought about by insisting upon requirements which have no valid relationship to the service conditions.

In conclusion, it is noteworthy that some very excellent theoretical research work has been done on the characteristic corrosion reactions of the stainless steels, but a great deal more is needed before the mechanisms of stress corrosion cracking can be understood or before adequate palliative measures can be established. On the other hand, there is perhaps an even greater need for good field corrosion work, because data are so difficult to transpose. It is essential that actual problems be studied in the field by corrosion men who understand the effects of thermal, mechanical and chemical treatment of the metal. Probably the greatest gaps in knowledge result from this dearth of good technical me nworking at plant level.

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DISCUSSION

By John A. Manning, Celanese Corporation of America, Charlotte, North Carolina.

The author implies a possible superior resistance of Type 329 stainless steel and of highly austenitic stainless steels. Recent tests of ours in a severe stress corrosion cracking environment consisting of methylene chloride-steam at 220 F indicates no significant difference in resistance to attack among:

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316
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317
309
310
321
329
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Only alloy Carpenter 20 and Nionel show freedom from cracking but are susceptible to pitting attack.

The author mentions the effect of oxygen and inhibitors and may wish to include as a reference: Stress Corrosion of Austenitic Stainless Steels in High Temperature Waters. W. Lee Williams and John F. Eckel. *ASNE Journal*, Pages 93-104 (1956) Feb. which indicates highly beneficial results from oxygen scavenging.

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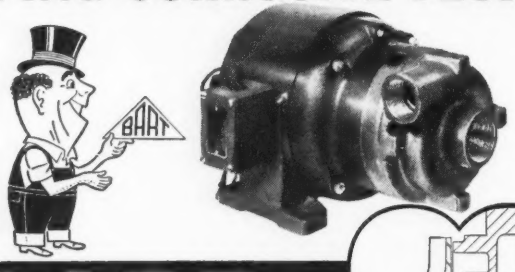

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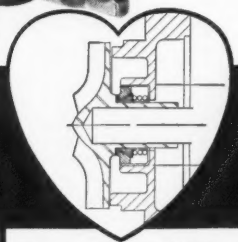

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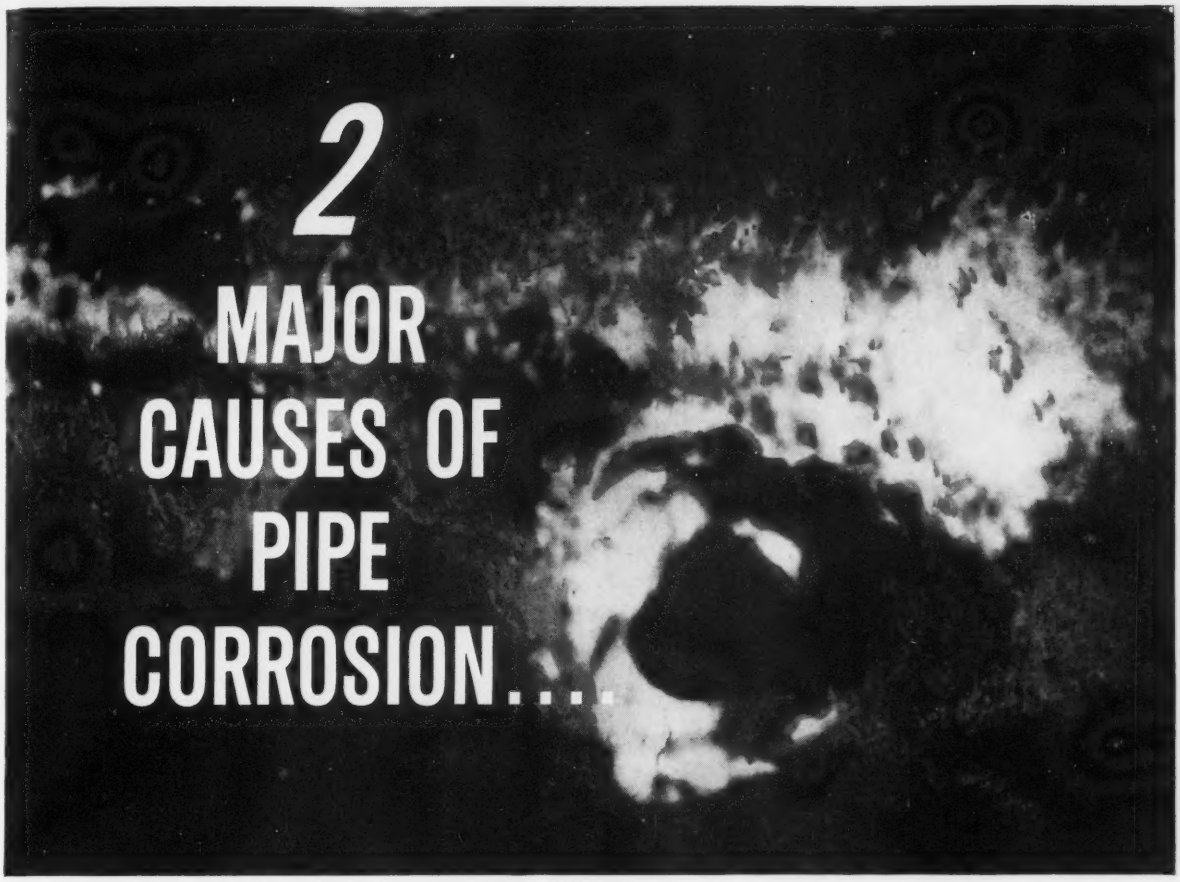
Chemical Surface Preparation of Structural Steel After Erection by Christopher D. Coppard, John H. Lawrence and Henry W. Adams.

Epoxy Coatings Perform Well on Steel Exposed to Chemicals, Corrosive Atmospheres by William M. Brackett.



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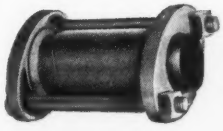
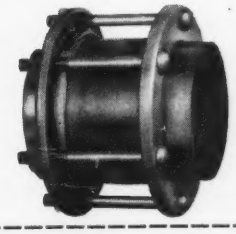
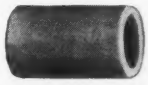


1¼" ID Style 39, showing two-bolt construction, supplied in sizes thru 2".



6" ID Style 39, showing construction supplied for this size. Note insulating gasket.



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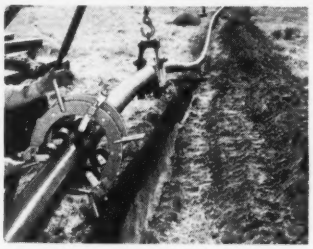

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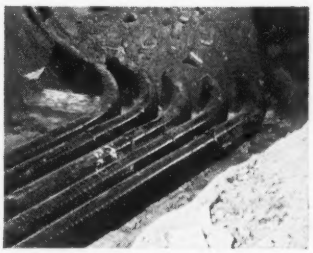

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CORROSION ABSTRACTS

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Vol. 13 October, 1957 No. 10

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1. GENERAL

1.6 Books

1.6, 3.1, 5.1

Corrosion—A Symposium. Book, 1956, 609 pp. The Committee of the Symposium on Corrosion (H. K. Worner, Chairman), Metallurgy School, University of Melbourne, Carlton N. 3, Victoria, Australia.

Papers presented during the symosium held at the Engineering School, University of Melbourne, November 28-December 2, 1955.

The book is divided into three sections: Fundamentals, Prevention and Corrosion in Industry. The first section includes 10 articles on various problems related to fundamental corrosion processes and testing methods. The section on prevention includes eight papers on coatings, inhibitors, cathodic protection, ship corrosion and external corrosion of gas mains and utilities. The third section includes papers on aircraft, chemical, gas, paper, petroleum and power industry corrosion problems. 13020

2. TESTING

2.1 General

2.1.1, 3.5.8, 1.6

Effect of Fatigue Stress History on Elasticity Properties and Stress Distribution Under Rotating Bending. L. C. LIDSTROM AND B. J. LAZAN. University of Minnesota. U. S. Wright Air Development Center, August, 1956, 55 pp. Available from: Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 121523).

An analytical method is described for determining the actual or specific stress-strain relationship from the moment-strain data of rotating beam fatigue specimens. The method is used for calculating the specific stress-strain relationships for mild steel. Data are presented on the effect of fatigue stress amplitude on the stress-strain properties of mild steel under axial (tension-compression) stress. The significance of the changes in modulus properties on the stress distribution in a rotating beam are discussed. Errors associated with nominal stress based on the accepted linear stress-strain relationships are also analyzed. Fatigue data on solid and hollow rotating beams under axial stress show poor agreement (12 to 30%) when compared on the basis of nominal stress. When compared on the basis of specific or true stress, determined by the methods given in the report, agreement is good (3 to 7%). 12947

2.3 Laboratory Methods

2.3.2, 4.2.7, 5.4.5

Tropical Deterioration of Automobile Paint Finishes. A. J. BIRCH. *Product Finishing,* **9,** No. 1, 50-64 (1956).

The behavior of car finishes during tropical exposure in S. Africa (Durban and Salisbury) and at Singapore is compared with results obtained in the author's accelerated weathering appartus. The performance of these paints varies markedly with climate; the accelerated weathering test appears to reproduce the behavior in the hot, wet Singapore climate. A special durability test for wood finishes which involves irradiation under humid conditions through a glass screen is described. Tests of the effect of plastic car covers on blistering and of stoving in contact with rubber are also discussed.—RPI. 12733

2.3.2, 5.4.5

Laboratory Studies on Anticorrosive Paints. M. TARBOURIECH. *Peintures, Pigments, Vernis,* **32,** No. 6, 506-514 (1956).

An accelerated test cycle for rust-inhibitive paints comprises immersion in running water, subjection to —15C to steam, to sulfur dioxide and to ultra violet radiation in moist air, in each case for 30 minutes. The paints are coated on both steel and glass panels, and the weight changes with time followed. Comparisons of the curves permits conclusions on the inhibitive properties of pigments and media. A collection of data is given relating to alkyd, bituminous and emulsion media, mica, lead cyanamide, red lead and zinc dust pigments.—RPI. 13413

2.3.7

Measurement of the Thickness of Thin Films by Multiple-Beam Interference. C. WEAVER AND P. BENJAMIN. *Nature,***177,** No. 4518, 1030-1031 (1956) June 2.

The film, the thickness of which is to be measured, is deposited on a substrate, part of which is covered to give a sharp edge to the film. An opaque metallic reflecting layer is then deposited over the edge of the film. The step in this overlayer is measured by using it as one surface of an interferometer and viewing the multiple-beam fringe system in reflection. Provided that the overlayer assumes the exact contours of the surface this stepheight will give the thickness of the film underneath. Experiments were carried out on the measurement of the thickness of thin silver films using both silver and chromium as the overlayer material. Illustration.—ALL. 12639

2.3.7, 2.4.2

Corrosion-Fatigue Testing in Highly Corrosive Media. W. P. MCKINNELL, JR., F. H. BECK AND M. G. FONTANA. *News in Eng.* (Ohio State Univ.), **28,** No. 4, 28-32 (1956) Nov.

Description of corrosion-fatigue testing machine. Graph shows corrosion-fatigue of titanium and 17-7 PH stainless in white fuming nitric acid at room temperature. Complete corrosion-fatigue studies of materials can be made with apparatus, taking into consideration the variables of testing speed and solution temperature as well as solution composition and strength. Diagrams.—INCO. 13294

3. CHARACTERISTIC CORROSION PHENOMENA

3.5 Physical and Mechanical Effects

3.5.8, 6.4.2, 4.2.4

Failure of Aluminium Alloy Fan Blades. J. P. HUGO. *S. African Mech. Eng.,* **5,** No. 9, 371-381 (1956) April.

Investigation of the failure (after 15 months' use) of cast aluminum-5 silicon ventilation fan blades operated at 580 rpm. Metallographic examination and other tests showed that the basic cause of failure was corrosion fatigue resulting from the presence of mine water in the atomosphere.—BNF. 13106

3.5.8, 7.6.4

Report of the Investigation of Two Generator Rotor Fractures. C. SCHABTACH, E. L. FOGLEMAN, A. W. RANKIN AND D. H. WINNE. Paper before Am. Soc. Mech. Engrs., Power and Metals Eng. Div., Ann. Mtg., Chicago, November 13-19, 1956. *Trans. ASME,* **78,** No. 7, 1567-1581; disc., 1581-1584 (1956) Oct.

Report of extensive investigation of recent fractures of the Arizona and the Cromby generator rotors. Nature and circumstance of burst, forging manufacture and properties, metallurgical examination of rotor material, and material-fracture characteristics are discussed for each case. Rotors were made from high-strength 2.50 nickel-0.40 molybdenum-0.03 vanadium steel. Results are given of notched-bend tests and relationship of notched-bend and Charpy V-notch impact tests are discussed. Both bursts were caused by unusual and severe stress concentrations combined with material characteristics. In Arizona rotor stress concentrations were created by numerous internal cracks in body of forging. In Cromby rotor, stress concentrations were caused by row of holes drilled and tapped in

rotor body for attachment of repair studs. Alloy segregation contributed to cause of both bursts. Hydrogen content of rotors lowered ductility significantly but was not the cause of brittle nature and rapid propagation of fractures. Tables, graphs, macro- and micro-photographs. —INCO. 13057

3.5.8

Fracture in Metals. N. F. MOTT. *J. Iron Steel Inst.*, **183,** No. 3, 233-243 (1956) July.

Ninth Hatfield Memorial Lecture, May 16th, 1956. Author discusses in detail ductile and brittle fracture in terms of dislocation theory, the latter with reference to ferrous metals and fatigue. 55 references.—BNF. 12838

3.5.8

Full Scale Fatigue Tests of Diesel Engine Elements. P. E. WIENE. *Trans. Inst. Marine Engrs.*, **68,** No. 3, 39-46 (1956) March.

Although this paper deals exclusively with experience with steels and cast irons there is much useful information on the effects of stress-raisers and the methods which can be used to mitigate these effects.—BNF. 12646

3.5.8, 1.6

Colloquium on Fatigue, Stockholm, May, 1955 (International Union of Theoretical and Applied Mechanics [IUTAM]). W. WEIBULL AND F. K. G. ODQVIST, Editors. Book, 1956, 339 pp. Published by Springer-Verlag, Berlin.

At this symposium, thirty-five papers were read from twelve countries; these are now reprinted with discussion (19 of the papers are in English, 7 in French and 9 in German. The discussions are given in each case in the language of the speaker). Reference to the book is greatly facilitated by subject indexes in English, French and German. The book production and illustrations are of the usual high quality associated with the Springer establishment.—BNF. 12635

3.5.8, 2.3.7

Brittle Fracture Propagation in Wide Steel Plates. W. J. HALL, R. J. MOSBORG AND V. J. MCDONALD. Paper before Am. Welding Soc., Nat'l. Fall Mtg., Cleveland, October 8-12, 1956. *Welding J.*, **36,** No. 1, 1s-8s (1957) Jan.

Structural grade rimmed steel plates were tested under various conditions of stress and temperature (room to 0 F) with brittle fracture initiated at an edge notch by wedge subjected to impact. Specimens were instrumented to provide record of strain response and crack speed as brittle fracture crossed specimen. Fracture initiation studies (explosive and impact methods) and instrumentation are described. Striking tests on specimens which did not fracture, indicate that small strain changes were produced by notch-wedge-impact method. Test records for 2- and 6-foot wide plate are discussed. Observations on strain response, crack speed and fracture appearance are presented.—INCO. 13476

3.5.8, 2.4.3, 6.2.3

Fatigue of Carbon Steel by Ultrasonic Flaw Detection Method. H. YAMANOUCHI AND T. INUKAI. *Repts. Castings Res. Lab., Waseda Univ.*, No. 7, 55-58 (1956) Oct.

Reports results of fatigue progress in carbon steel using an ultrasonic flaw detector. Values of ultrasonic attenuation constant in mild steel under fatigue vary with repeated stress intensity and stress cycle; values increase in proportion to stress cycle but transition point exists on the way.—INCO. 13449

3.6 Electrochemical Effects

3.6.2, 4.6.7, 6.2.2

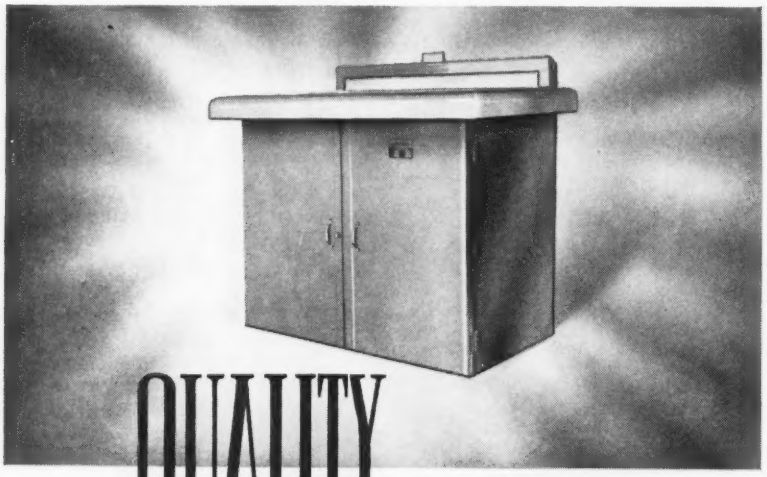
Tuberculation of Tar-Coated Cast Iron in Great Lakes Water. THURSTON E. LARSON, R. V. SKOLD AND E. SAVINELLI. *J. Am. Water Works Assoc.*, **48,** Pt. 1, 1274-1278 (1956) October.

Electrical measurement; roughness; galvanic effect.—BTR. 13160

3.6.5, 3.8.2

The Surface Potential of Metals and the Galvani Potential of Metal/Solution. (In German.) PAUL RUETSCHI. *Z. Elektrochem.*, **60,** No. 1, 29-31 (1956).

The Galvani potential difference between two phases $\Delta\phi$ is the sum of the Volta potential difference $\Delta\psi$, a measurable quantity, and the surface potential difference $\Delta\chi$. A method is given for finding the surface potential difference by applying a theoretical thermodynamic cycle in which a molecule of alloy metal is separated by infinite dilution, evaporated, ionized, the positive ions and electrons returned to the alloy and in the alloy reunited to equilibrium. From this the exit energy of foreign metal ions $\alpha_i = -A + S + J - \phi - J'$, where A = mixing energy, S = sublimation en-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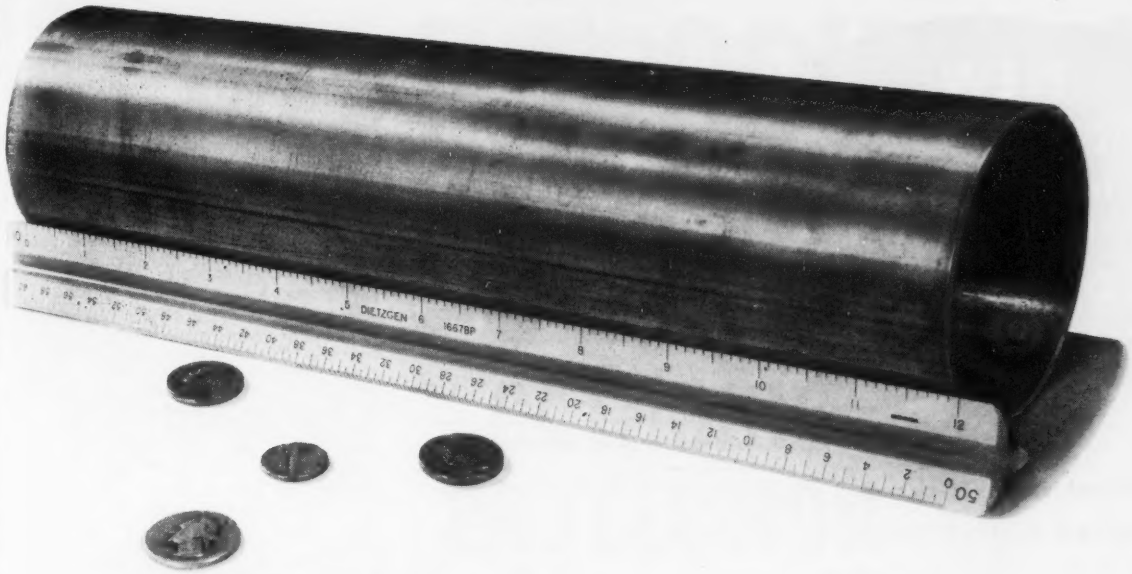

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ergy, J = ionization energy, ϕ = electron-exit work from the solution, J′ = ionization work in the alloy. For alkali metals the further equation has been derived—

$$\alpha_i = \frac{C_1}{r} + \frac{C_2}{r^2} - P,$$ where r = atomic radius of added element, C_1 and C_2 are constants and P = surface potential. Using known values for lithium, sodium, potassium, rubidium and cesium, for the case of dilute amalgams the surface potential of mercury is calculated. It is found to be probably <0.2 v. and the Galvani potential of mercury/solution is not far from zero. The Galvani potential difference mercury/solution against a N-calomel electrode is found to be between 0.5 and 0.7 v. 18 references.—MA. 13371

3.7 Metallurgical Effects

3.7.3, 2.3.7

Development of a Ferritic-Austenitic Weld Joint for Steam Plant Application. J. T. Tucker, Jr. and F. Eberle. Paper before Am. Soc. Mech. Engrs. Metals Eng. Div. Conf.—Am. Welding Soc. Nat'l. Spr. Mtg., May 7-11, 1956. *Welding J.*, **35**, No. 11, 529s-540s (1956) Nov.

Thermal-cycling tension test gives realistic indication of dissimilar weld behavior in tubing and piping under cyclic conditions of temperature and stress in steam plant service. Development of the test and study of factors affecting dissimilar weld life are discussed with reference to ferritic-austenitic weld junctions of 2¼ chromium-1 molybdenum steel joined to 18-8-niobium with 19-9-niobium electrodes. Holding time at temperature (1100 F), stress-oxidation at constant temperature (1100 F), cyclic stressing in absence of oxygen, thermal cycling from lower temperature (1050 F) and from higher temperature (1200 F) and effect of postweld heat treatment are factors considered. Satisfactory welds between 2¼ chromium-1 molybdenum steel and 18-8-niobium, capable of withstanding cyclic conditions of temperature and stress, can be made by eliminating high thermal-expansion stresses at ferritic-austenitic junction with an austenitic weld metal

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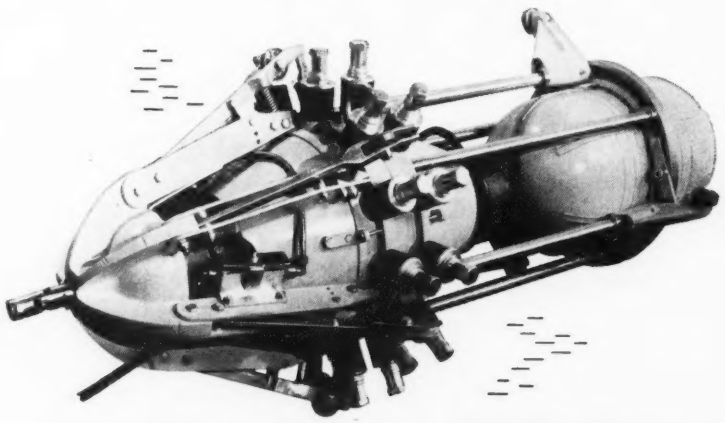

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3.7.3, 3.2.2

Hydrogen Embrittlement of Cold-Worked Metals. R. L. MILLS AND F. J. EDESKUTY. Univ. of California. *Chem. Eng. Progress,* **52,** No. 11, 477-480 (1956) Nov.

Types 303, 316 and 347 stainless, carbon steel and beryllium-copper were examined for effect of prior cold-working on susceptibility to room-temperature hydrogen embrittlement. Carbon steel cold-worked to 25-45% reduction in area was permanently embrittled by absorption of cathodic hydrogen. Presence of cold-formed ferrite in austenite does not significantly lower resistance of stainless to hydrogen embrittlement. Tables, diagrams.—INCO. 13272

3.7.3, 3.2.2

On the Nature of Embrittlement Occurring While Tempering a Nickel-Chromium Alloy Steel. G. BHAT AND J. F. LIBSCH. Paper before AIME, Cleveland, Oct., 1956. *J. Metals* (Trans. AIME), **9,** No. 1, 20-22 (1957) Jan.

Study of characteristics of two modes of embrittlement proposed in commercial nickel-chromium steel (AISI 3140). Data are presented that show that embrittlement occurring at 925 F may be removed completely by reheating for short times and has no influence upon further embrittlement at 1250 F. Embrittlement at 1250 F appears related to permanent structural change which has significant retarding influence upon subsequent development of embrittlement at 925 F. It is suggested that segregation of solute atoms to prior austenite and ferrite grain boundaries may provide explanation for two modes of embrittlement. Transition temperature data are plotted down to —120 F.—INCO. 13195

3.8 Miscellaneous Principles

3.8.2, 6.3.19, 3.7.4

Electrode Potentials of Zinc Single Crystals in Aqueous Solutions. N. S. AKHMETOV AND G. S. VOZDVIZHENSKII. *J. Applied Chem., USSR* (Zuhr. Priklad. Khim.), **29,** No. 8, 1197-1202 (1956).

A relationship is established between the potential and crystal face orientation. 6 figures, 20 references.—ATS. Translation available from: Associated Technical Services, P. O. Box 271, East Orange, New Jersey. 12770

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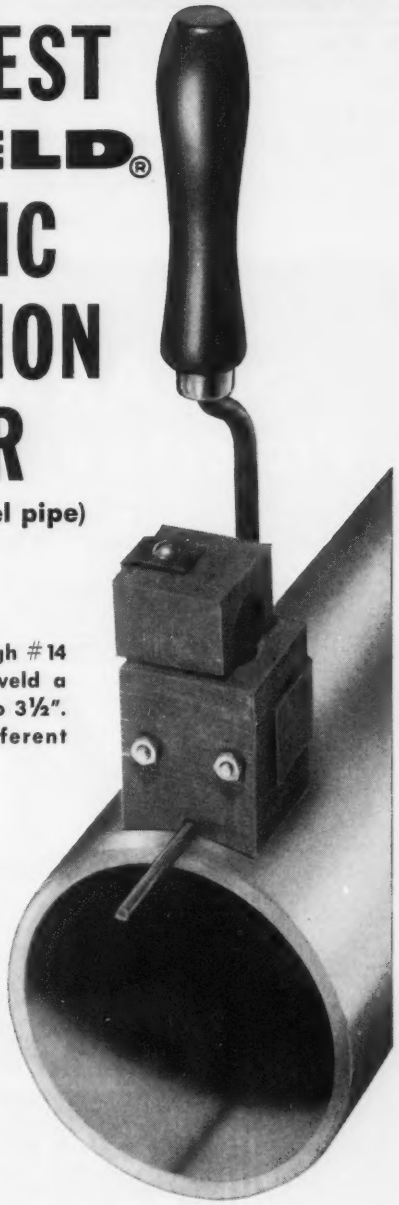

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3.8.3, 6.2.2

On the Nature of the Passive Film on iron in Acid Solutions. (In Russian.) A. M. SUKHOTIN. *Uspekhi Khimii* (Progress in Chem.), **25,** No. 3, 312-328 (1956).

3.8.3, 6.3.3

Studies in the Passivity of Chromium. I. The Electrodic Behavior of Chromium and Its Bearing on the Passivity of the the Metal. I. M. ISSA AND H. KHALIFA. *J. Indian Chem. Soc.*, **33,** 465-474 (1956) July. 13176

3.8.3, 6.3.10

Passivity of Nickel: Anodic Behavior of Nickel-III. (In German.) G. TRUMPLER AND W. SAXER. *Helv. Chim. Acta*, **39,** No. 6, 1733-1741 (1956).

Passivation for pure nickel above a certain potential barrier. Behavior of current in transition from an active to a passive state and at various potential levels.—BTR. 13424

3.8.4

An Attempt at a Theoretical Interpretation of the Solubility of Metals in Water. (In French.) JEAN-CHARLES PARIAUD AND PIERRE ARCHINARD. *J. Chim. Phys.*, **53,** No. 9, 765-769 (1956) Sept.

Study of a limit of solubility and of its causes. Application of the Nernst equation for various metals.—MR. 13332

3.8.4

Stability of Ferrous Hydroxide Precitates. F. J. SHIPKO AND D. L. DOUGLAS. *J. Phys. Chem.*, **60,** 1519-1523 (1956) Nov. 13372

3.8.4, 3.2.3

Formation of Composite Scales Consisting of Oxides of Different Metals. CARL WAGNER. *J. Electrochem. Soc.*, **103,** 627-633 (1956) November.

A theoretical analysis of diffusion processes during the oxidation of an alloy when oxides of different alloying elements are formed concurrently.—BTR. 13428

4. CORROSIVE ENVIRONMENTS

4.6 Water and Steam

4.6.2

Corrosion in Condensate Systems Can be Controlled. R. G. DALBKE. Dearborn Chem. Co. *Power Eng.*, **60,** No. 8, 89-91, 134 (1956) August.

Oxygen and carbon dioxide dissolved in water are primary causes of corrosion in condensate systems. Oxygen-type corrosion is characterized by pitting of metal walls and can be handled by use of a well vented deaerating heater followed by oxygen scavenging chemicals in boiler water. Carbon dioxide is the most common cause of return-line corrosion causing a general thinning or grooving of metal walls. Two basic chemical methods used to control corrosion in return systems are ammonia and amines to neutralize dissolved gases and use of inhibitor amines which form a protective film on metallic surfaces. Several case histories of condensate corrosion are given. Diagrams, photos.—INCO. 12743

4.6.2, 2.3.4, 6.2.3

The Corrosion of Mild Steel by High Temperature Water. J. N. WANKLYN. Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England, Rept. M/M-116, May, 1956, 8 pp.

Methods of measuring and expressing the corrosion of carbon steel by high temperature water are discussed. Recent information on the influence of a number of factors is summarized. (auth).—NSA. 13414

4.6.2, 5.7.3

The Use of Hydrazine as an Oxygen Scavenger in H.P. Boilers. W. F. STONES. *Chemistry and Industry*, No. 5, 120-128 (1957) Feb. 2.

Discussion of properties and handling precautions of hydrazine. Study of hydrazine as a feed water deoxidant was carried on in boilers whose condenser tubes and feed heaters were of 70-30 copper-nickel and brass. Addition of hydrazine 100% in excess of the oxygen figures, results in a fairly rapid rise in ammonia concentration and thus an increase in pH of the condensate. Consideration of results for ammonia, oxygen and hydrazine leads to conclusion that once a hydrazine residual is established in boiler, most of the excess hydrazine decomposes into ammonia and that boiler residual for a given pressure is dependent on a rise in oxygen. Therefore an increased quantity of excess hydrazine results in a slightly higher hydrazine residual and a rise in the ammonia concentration in the steam and condensate. Determination of copper and iron in feed water is described and comparative costs of hydrazine and sulfite treatment are given. Graphs, diagrams, tables.—INCO. 13471

4.6.2, 5.8.2, 5.7.3

Re-Use of Steam Condensate as Boiler

Feedwater. D. E. NOLL AND H. M. RIVERS. Hall Labs., Inc. *Ind. & Eng. Chem.*, **48**, No. 12, 2146-2150 (1956) December.

Modern pretreating facilities, efficient deaerating equipment, proper materials of construction, devices for policing system and chemicals for conditioning steam make possible effective re-use of condensate as boiler feedwater. Description of mechanism of corrosion by low pH, carbon dioxide and oxygen in condensate systems and deposition of corrosion products of iron and copper oxides is given. Use of sodium sulfite and hydrazine as oxygen scavengers and steam conditioning by addition of ammonia or neutralizing and filming amines are discussed. Diagrams, photos. 14 references.—INCO. 13303

4.6.2, 7.6.4

Water Treatment for Boilers: Natural Controlled Circulation, Once-Through. R. C. ULMER. Paper before Penna. Elec. Assoc., Fall Mtg., Hicksville, L. I., October 18, 1956. *Combustion*, **28**, No. 6, 61-66 (1956) Dec.

Discussion of design of modern boilers with emphasis on general circulation and problems and goals for water treatment. Preboiler corrosion leading to deposits is due to low pH and/or dissolved oxygen in the preboiler system. Other types of attack are general and localized, or pitting. Acid cleaning eliminates present deposits; new ones are eliminated through use of proper preboiler treatment. Prevention of boiler corrosion by pH control, maintaining dissolved oxygen at a low value and maintaining an excess of reducing agent, sodium sulfite or hydrazine in the system is discussed. Photomicrographs, graphs, tables, 19 references.—INCO. 13363

4.6.4, 3.3.4, 5.7.9

Biological Fouling in Recirculating Cooling Water Systems. J. J. MAGUIRE. W. H. & L. D. Betz. *Ind. & Eng. Chem.*, **48**, No. 12, 2162-2167 (1956) Dec.

Discussion of use of chlorine, bromine, chlorinated phenols, copper salts and quarternary ammonium compounds to prevent interference with heat transfer caused by slime and algae in recirculating cooling water systems. Photos, tables. —INCO. 13290

4.6.4, 7.4.1, 6.6.11

A Case History in Cooling Water Treatment. C. J. HOLLAND, JR. AND W. A. TANZOLA. *Corrosion*, **13**, No. 4, 257t-262t (1957) April.

The problems of corrosion and tower wood deterioration are commonly encountered in recirculation cooling water systems. At the Delhi Natural Gasoline Plant in Louisiana the concurrent presence of these two problems resulted in plugging of heat exchanger tubes and in high metal loss throughout the system. The corrosive potential of the water was aggravated by "fiber throw" in the water caused by serious deterioration of the tower wood. The "fiber throw" enmeshed with corrosion products created cell action which produced serious pitting and tuberculation.

A program of treating the water was undertaken both to minimize the attack of the wood and to prevent corrosion. Effectiveness of the treating program was followed by corrosion studies to evaluate the benefits of each step taken. Initially, rather high treatment concentrations were necessary to keep corrosion under control. But as the clean-up program to aid in freeing the system

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of corrosion products and fiber took effect, the corrosion load was reduced, which permitted good control of corrosion with normal treatment concentrations. 13445

4.6.5, 6.4.2

Corrosion of Type 2S Aluminum in Distilled Water. G. M. INMAN AND J. J. SHYNE. North American Aviation, Inc., Downey, Calif. U. S. Atomic Energy Comm. Pubn., NAA-SR-Memo-26, April 10, 1951 (Declassified April 13, 1956), 15 pp. Available from: Office of Technical Services, Washington, D. C.

Corrosion of 2S aluminum in contact with distilled water at room temperature presented an unexpected and serious problem during experiments with an exponential assembly. Effective coatings were developed for the aluminum tubing surfaces affected, which could provide protection against corrosion up to at least 80 C. The coatings have safely low neutron absorption cross sections. The probable mechanism of corrosion is briefly discussed. (auth).—NSA. 13179

4.6.6

Water Treatment for Urban Buildings. S. SUSSMAN. Water Service Labs., Inc. *Consulting Engr.*, **9**, No. 1, 71-74, 76, 78 (1957) Jan.

Most public water supplies along heavily populated Atlantic Seaboard are corrosive. With ferrous piping, including galvanized iron and steel, an unsightly rusty water condition can develop within 6-12 months in hot water systems, followed by clogging of pipes and development of leaks in pipe wall. Two methods used for protection of piping systems in urban buildings are neutralizing filters and addition of soluble, nontoxic corrosion inhibitors. Magnesium anodes sometimes are used for preventing corrosion of certain pieces of water handling equipment in buildings and are effective with waters of high mineral content. Treatment methods are recommended for air conditioning units, swimming pools, sprinkler systems and hydraulic elevators. Photos, tables, 25 references.—INCO. 13183

4.6.8, 7.3

Corrosion Problems in Pumping Acid Mine Water. C. D. CLARKE AND G. REINBERG. Paper before Am. Inst. Mining Met. Engrs., New York Mtg., Feb., 1956. *Mining Eng.*, **8**, No. 8, 821-825 (1956) August.

Stray currents that cause corrosion are direct current and are encountered in mines using direct current haulage systems. Acid mine waters are good electrolytes and provide favorable environment for galvanic corrosion. Insulating pump adequately from stray currents also insulates it from possibility of action from adjoining and connected equipment. Galvanic action within the pump cannot be controlled; equipment must be entirely of similar metals if such corrosion is to be avoided. Diagrams.—INCO. 12757

4.7 Molten Metal and Fused Compounds

4.7, 6.3.10, 3.5.3

The Corrosion and Erosion of Nickel by Molten Caustic Soda and Sodium Uranate Suspensions Under Dynamic Conditions. J. N. GREGORY, N. HODGE AND J. V. G. IREDALE. Gt. Brit. Atomic Energy Res. Establishment, Harwell, Berks, England, Rept. C/M-273, March, 1956, 24 pp.

Compared with that measured under static conditions, the corrosion rate of nickel in molten caustic soda under dynamic conditions is approximately 10 times as great. The addition of sodium uranate increases this further by a factor of 3 at 600C although at 500C the uranate has no effect. The operation of thermosyphon loops containing molten caustic soda has revealed some of the difficulties and limitations of the system. Under an inert gas atmosphere at temperatures above 600C mass transfer of nickel causes complete obstruction of the flow in a few weeks, while at 500C, blocking occurs after about 200 days. In these cases the mass transfer deposit is a higher oxide of nickel whose composition is complex, but approximates to nickel sesquioxide. Under a hydrogen atmosphere, loops are slower to plug than those operated under an inert atmosphere at the same temperatures and here the deposit is found to be pure nickel. (auth) —NSA. 12803

4.7

Corrosion and Components Studies on Systems Containing Fused Sodium Hydroxide. EUGENE M. SIMONS, NEIL E. MILLER, JOHN H. STANG AND C. VERNON WEAVER. Battelle Memorial Inst. U. S. Atomic Energy Comm. Pubn., BMI-1118, July 30, 1956, 46 pp. Available

CONTINUOUS LABORATORY CHECKS OF INGREDIENTS AND PROCESSES RESULT IN MAINTAINING INTERNATIONAL MARINE COATINGS AT THE HIGHEST STANDARDS

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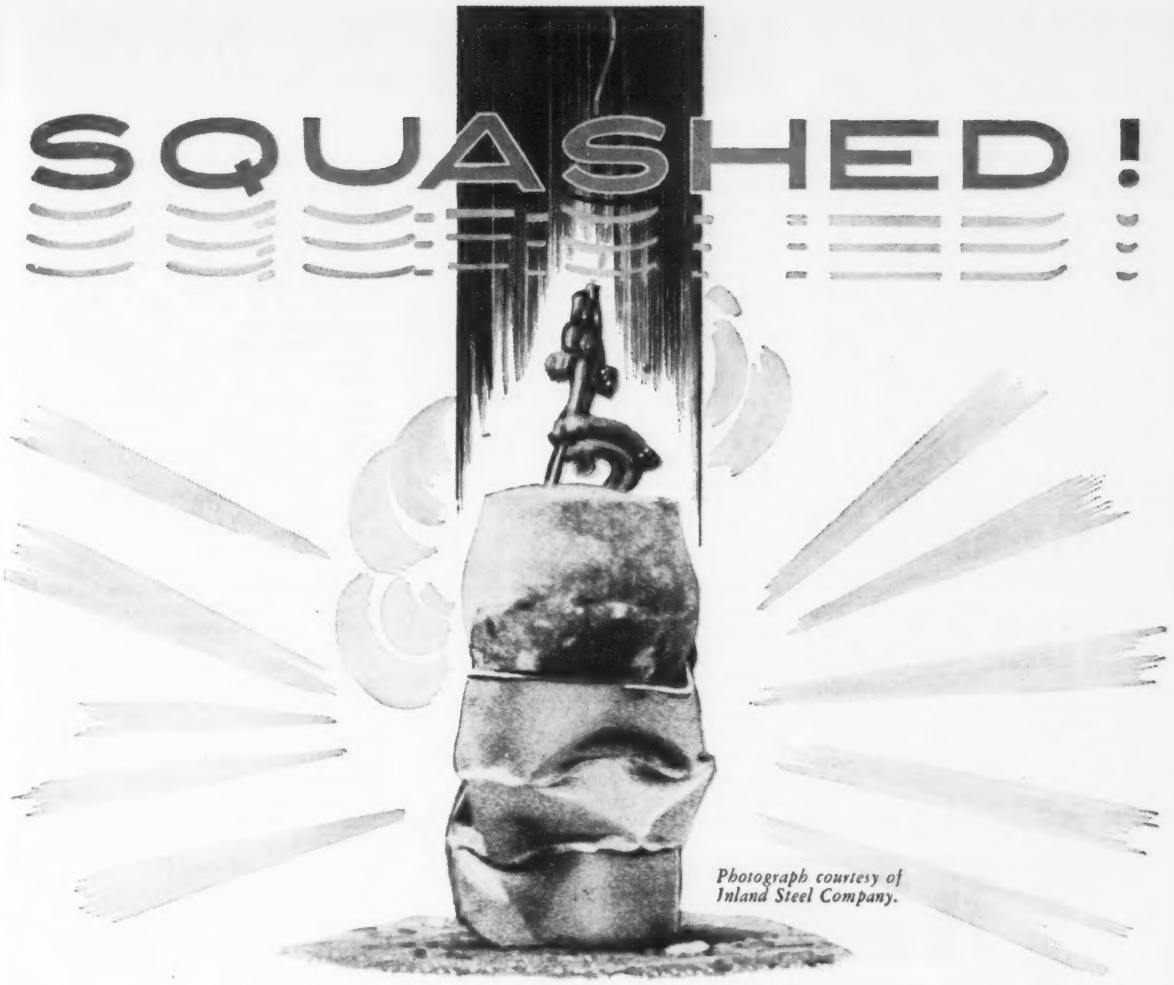

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Information is presented from a group of experiments in which the corrosiveness of fused sodium hydroxide (900 to 1500 F) was examined. Cold zones of all nickel natural- and forced-convection loops in which hydrogen-blanketed sodium hydroxide was circulated at peak temperatures of 1200 to 1500 F with temperature cycles in the range of 100 to 200 F plugged severely by nickel deposition in times ranging from less than 10 to 200 hours. In 900 and 1050 F peak-temperature experiments with all nickel pumping-loop systems, considerably better results were obtained. For example, after 2000 hours of operation, a 900 to 70 F thermal-cycle system displayed a cold-zone metal transfer of only 5 mils. This result indicates that, in this temperature range, a reasonable life for a sodium hydroxide flow system is possible, although a relatively low fluid-temperature cycle would be required. Several corrosion inhibitors evaluated in static capsule experiments failed to depress the corrosiveness of 1500 F sodium hydroxide. One additive, magnesium oxide, appeared to inhibit attack noticeably but its effect was far from enough to eliminate the problem. In addition to the corrosion work, experiments were conducted to evaluate components requirements for sodium hydroxide flow systems. These indicated that the straightforward engineering approaches to components problems in large-scale flow systems would be generally successful, providing the materials problems were solved. There would, however, be a special need for extensive work on components that would require pressure-sensing elements and those that would function as seals, both static and dynamic. Frozen-type seals, which hold some promise for the latter application, received considerable attention during the experimental phases of this work. (auth). —NSA. 13380

4.7, 3.5.8

A Preliminary Study of the Fatigue of Metals in Liquid Metal Environments. J. W. Martin and G. C. Smith. *Metallurgia*, **54**, No. 325, 227-232, 238 (1956) November.

Two types of fatigue tests were carried out. First series were rotating bending tests, using Wohler-type machine

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and Haigh-Robertson wire testing fatigue machine to determine effect of mercury coating on fatigue properties of 70-30 and 60-40 brasses, copper-tin alloy and copper-cadmium alloy. Second series were push-pull tests, using Losenhausen hydraulic machine, on specimens of mild steel in contact with liquid tin at 300 C and stainless steel (18-8) in contact with liquid sodium at 300 C. Liquid metal was contained within hollow test-pieces. Microphotographs show fatigue fracture in brass, penetration of liquid tin around sulphide inclusion in mild steel and transgranular cracks near fatigue fracture of 18-8.—INCO. 13283

5. PREVENTIVE MEASURES

5.4 Non-Metallic Coatings and Paints

5.4.8

Acid and Alkali Resistant Protective Paints. Part II. Coatings Based on Epoxide and Other Resins. W. L. YEO, *Corrosion Prevention & Control,* **3,** No. 1, 35-36 (1956) Jan.

Coatings based on epoxide resins provide a high standard of protection against acid attack and reasonable protection against alkalinity and a wide range of solvents. The air drying epoxide enamel is more frequently used. Such enamels have a full hard gloss and are not affected by prolonged exposure to ultra-violet and sunlight. Cold cured enamels have the advantage that they can be applied very thick because they polymerize without oxidation. Vinyl paint systems and silicone resin paints also perform well. —RPI. 12657

5.4.8, 4.6.5

Testing of Baking Enamels for Storage Tanks for Demineralised Boiler Feedwater. E. HASS. *Mitt. Ver Grosskesselbesitzer,* No. 40, 35-39 (1956); Chem. Absts., **50,** No. 13, 9758f (1956).

Baking enamels have good ageing properties, combined with superior adhesion, so that they are suitable for coating pipes and holders for protection from corrosion by cold and hot water. With the present high purity of steam condensate and water from ion-exchange systems, these lacquers should be tested for soluble constituents. Water standing in contact with them at 20 and 40 degrees for periods up to three weeks was tested for residue on evaporation, increase in potassium permanganate value and conductivity. One enamel was found to be unsatisfactory, but another yielded only negligible amounts of soluble material and its characteristics were unchanged over the testing period. These enamels were a mixture of high-polymer synthetic resins and a suitable silicone containing varying amounts of pigments. They were polymerized at about 180 degrees. —RPI. 13231

5.4.8, 4.6.11, 4.2.5

Suggested Painting Specifications for Marine Environments: First Interim Report. NACE Technical Unit Committee T-1M on Corrosion of Oil and Gas Well Producing Equipment in Offshore Installations. (D. F. DIAL, JR., CHAIRMAN), Prepared by Task Group T-1M-2 on Protective Barriers (H. E. WALDRIP, CHAIRMAN). *Corrosion,* **13,** No. 3, 205t-208t (1957) March.

Specifications and recommendations are given significant to contractors and operators applying and purchasing coatings systems in marine environment. The recommendations cover equipment, surface preparation, specific coatings systems, inspections and the techniques related to these items. 13140

5.4.8, 4.6.11, 4,2,5

Suggested Coating Specifications for Hot Application of Coal Tar Enamel for Marine Environments: Second Interim Report. NACE Technical Unit Committee T-1M on Corrosion of Oil and Gas Well Producing Equipment in Offshore Installations (D. F. DIAL, JR., CHAIRMAN), Prepared by Task Group T-1M-2 on Protective Barriers (H. E. WALDRIP, CHAIRMAN). *Corrosion,* **13,** No. 3, 209t-210t (1957) March.

Specifications and recommendations are given significant to contractors and operators applying and purchasing coal tar coatings systems in marine environments. Recommendations cover surface preparation, materials and systems, handling of materials, application and inspection. 13139

5.4.8, 8.9.5

Modern Marine Paints. A. D. C. HAMILTON. *Corrosion Technology,* **4,** No. 1, 5-8 (1957) Jan.

Discusses development in marine paint formulation resulting from new and improved raw materials. Various methods of surface preparation and paint application are reviewed. Paint compositions and their protective merits are discussed for bottom compositions, above-water primers, boot-top compositions, topside, superstructure, accommodation and cargo space paints. —INCO. 13184

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For the most profitable results, a pipe line crew *must* work as a team . . . and the same is true of the materials they use . . ."GLASFAB"and GLASKOTE comprise the *top* team in the pipe wrap league.

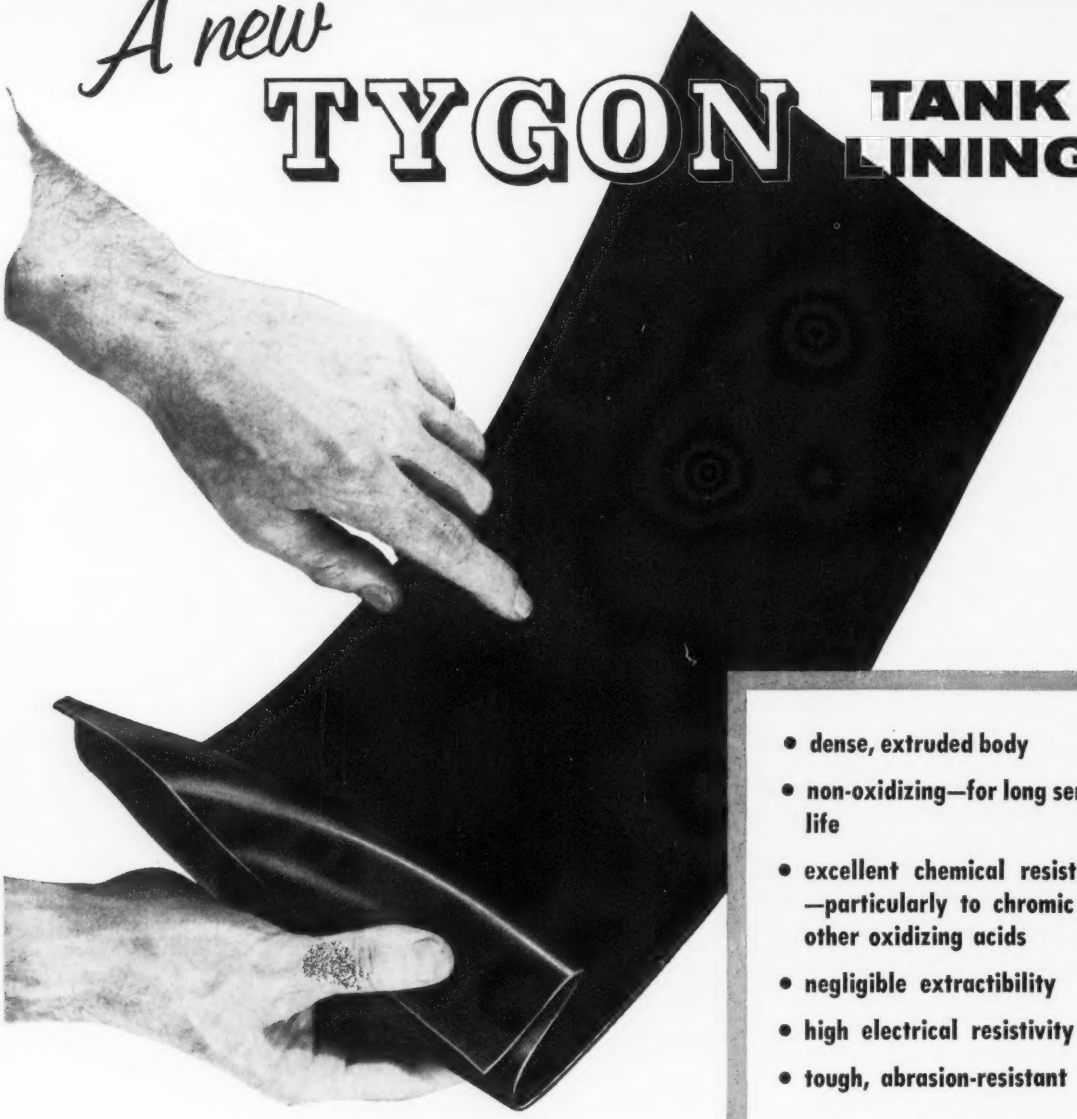
"GLASFAB". . a wholly inorganic, loom woven glass fabric pipe wrap, with maximum tensile strength, porosity, and reinforcing qualities. GLASKOTE . . . a *ready-to-apply* plasticized coal tar pitch and glass fabric in tape form with high resistance to soil stress, high impact resistance and a superior bonding strength . . excellent for emergency repairs.

Make your team more mobile and efficient . . . consult a Midwestern representative about"GLASFAB"and GLASKOTE TODAY.

branch offices—
• Houston, Tex.
• Atlanta, Ga.
• Mt. Prospect, Ill.
• Pittsburgh, Pa.
• Oklahoma City, Okla
• Portland, Ore.
• New York, N.Y. (export)

MIDWESTERN
PIPE LINE PRODUCTS CO.
4645 Sapulpa Rd. Tulsa, Okla. HIckory 6-6144
Cable Address: Mid Pipe

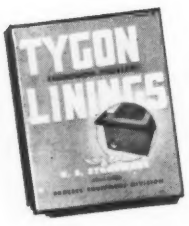
A new TYGON TANK LINING



- dense, extruded body
- non-oxidizing—for long service life
- excellent chemical resistance —particularly to chromic and other oxidizing acids
- negligible extractibility
- high electrical resistivity
- tough, abrasion-resistant

This new Tygon tank lining material (Formulation 105A) represents a major step forward in the development of plastic linings for corrosive service. This new lining is *extruded*, not calendered. It is completely free of laminations, hence no "peeling" in service. It shows excellent resistance to highly oxidizing acids, including chromic.

103-F



Tygon Tank Linings can be installed at our plant or in the plants of licensed fabricators throughout the United States and Canada. Write today for Bulletin TL-526R.

U. S. STONEWARE
AKRON 9, OHIO

210a

Now you can have

maximum protection

for every inch of underground piping with

Carey®

asbestos pipeline felt

only 4% weight variation in 30,000 miles of felt — thanks to new **AccuRay**® process!

This AccuRay control system has watch-dogged the manufacture of more than 30,000 miles of Carey Pipeline Felt. During this time the average weight variation in the felt has been less than 4%—compared to the usual 30% average for standard quality control methods.

® Trade mark of Industrial Nucleonics, Inc.—originators of this advanced nucleonic process for measuring thickness.

Every inch of underground pipe is subjected to soil stress. One weak spot in 100 miles of piping can be mighty costly—and so can any weak spots in the felt that protects this piping. That's why it's just good business to choose the pipeline felt that gives you the greatest uniformity of weight, thickness and strength for maximum protection against corrosion.

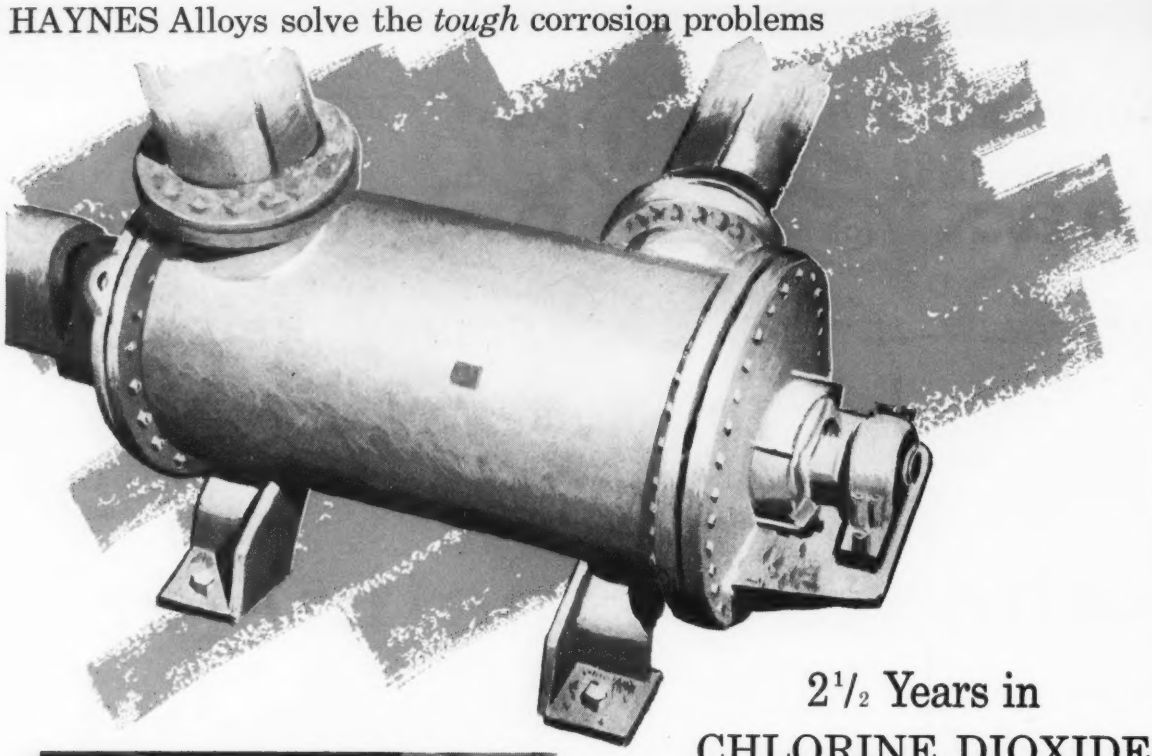
Only Carey, of all pipeline wrapping manufacturers, can offer you this uniformity—because only Carey Asbestos Pipeline Felt is weight-controlled by nucleonic AccuRay. There can be no *weak spots* to cause you repair headaches in years to come.

Why not ask your Carey Sales Engineer for the full story on the new Carey Asbestos Pipeline Felt with AccuRay, or write Dept. C-107 in Cincinnati.

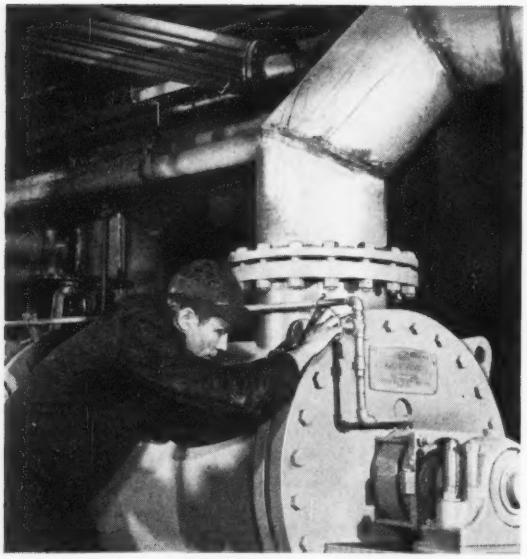
THE PHILIP CAREY MFG. COMPANY
Lockland, Cincinnati 15, Ohio

Manufacturers of
Carey Pipeline Felt, Pipeline Padding, and Carey Glass

HAYNES Alloys solve the *tough* corrosion problems



2½ Years in CHLORINE DIOXIDE ... and still operating



. . . Process Equipment Protected with HASTELLOY Alloy C . . . can be used to handle acids, fats, oils, or other corrosive materials. This vessel made of HASTELLOY alloy C handles chlorine dioxide used in a pulp bleaching process.

To give greater brightness to kraft paper, the pulp is bleached with chlorine dioxide (ClO_2). This creates a mixer and equipment problem—some of the mixer materials lasted only six months.

One mixer, lined with ⅛-in. HASTELLOY alloy C sheet, has lasted 2½ years—5 times longer than any previous material used. A mixer made entirely of HASTELLOY alloy C has a full life expectancy of 10 years.

If you have a severe corrosive condition, it will pay you to investigate the use of HASTELLOY alloys. Send for Booklet. Address HAYNES STELLITE COMPANY, Division of Union Carbide Corporation, Kokomo, Indiana.

HAYNES ALLOYS

HAYNES STELLITE COMPANY

Division of Union Carbide Corporation

UNION CARBIDE

"Haynes," "Hastelloy" and "Union Carbide" are registered trade-marks of Union Carbide Corporation.

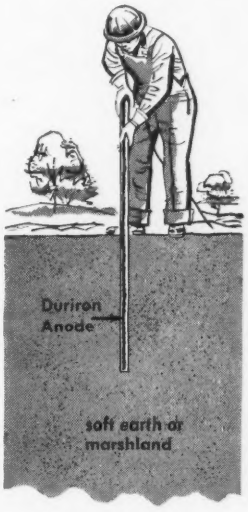
32 DURIRON® ANODES protect 11,000 feet of steel water line without backfill

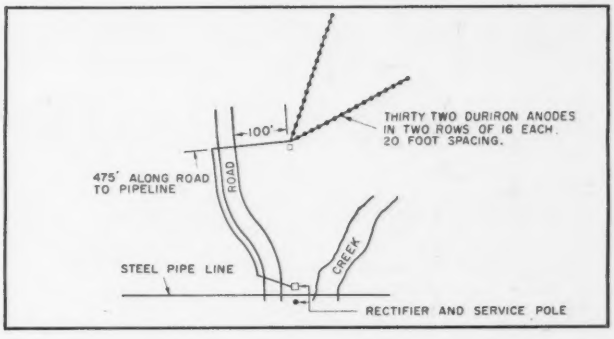
The Problem:

An isolated section of 11,000 feet of steel water line needed cathodic protection. Tests showed current requirements to be 40 amps., but replacement of adjacent wood stave lines called for extra capacity. Ground was a peat bog with water table standing at 6 inches below surface, making backfill methods impossible.

The Installation:

32 Duriron Anodes (2" x 60") were installed without backfill, by digging through the dry surface and then pushing anodes down until the tops were 3 feet below surface. Anodes were installed 20 feet apart, in two rows, with a calculated resistance of one ohm, based on previous experience with similar installations. While conventional backfill columns would have reduced the number of anodes necessary, the use of bare anodes constituted a major saving in cost.




100'
THIRTY TWO DURIRON ANODES IN TWO ROWS OF 16 EACH. 20 FOOT SPACING.
475' ALONG ROAD TO PIPELINE
ROAD
CREEK
STEEL PIPE LINE
RECTIFIER AND SERVICE POLE

The Performance:

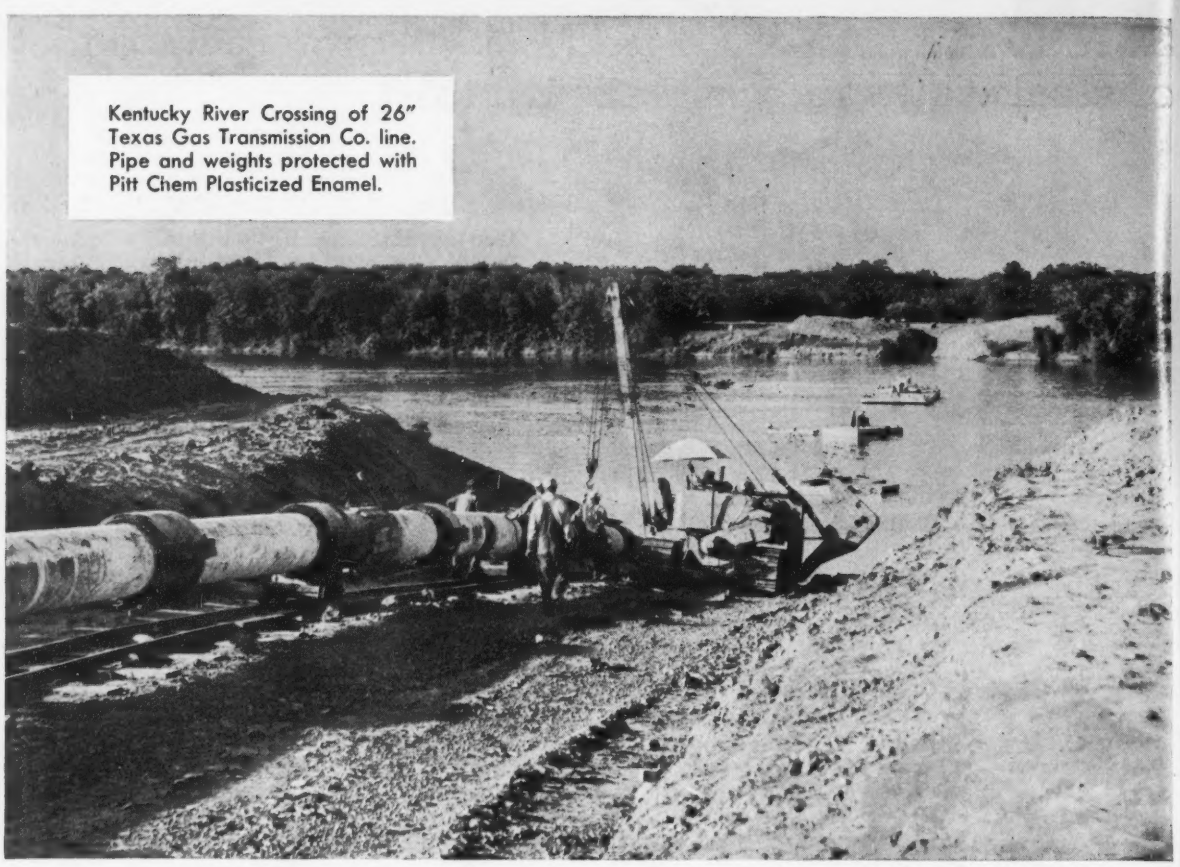
The anode bed is discharging 60 amps. at a terminal voltage of 59 volts. The entire line section is under protection, the minimum potential being 1.18 volts. This affords ample margin to handle prospective additions to the line. Agreement between actual and calculated anode resistance is quite close.

Action:

For further information on this particular installation or for technical assistance on other Duriron impressed current anode installations, write:

DURCO
CORROSION RESISTING ALLOYS & EQUIPMENT

THE DURIRON COMPANY, INC. / DAYTON, OHIO



Kentucky River Crossing of 26" Texas Gas Transmission Co. line. Pipe and weights protected with Pitt Chem Plasticized Enamel.

Pipeline Reconditioning Here Could Cost A Fortune!

But Pitt Chem Enamel Assures Long-Term Protection and Low Maintenance Cost

THE extra cost of protecting your pipelines with coal tar enamels may look like pin money a few years from now. For the expense of excessive cathodic protection and reconditioning—which is often necessary when "economy" coatings are used—can sky-rocket your maintenance costs out of all economic proportion.

Coal tar enamels have *proved* their superior ability to resist soil stress and water absorption, the two principal reasons why other coating materials repeatedly fail. So why gamble? Specify *Pitt Chem* Coal Tar Enamels, just as scores of leading pipeline companies do each year, and be sure of minimum maintenance and long-lasting protection.

When you specify *Pitt Chem*, you enjoy the advantages of guaranteed quality, for *Pitt Chem* Enamels are manufactured under rigid production control—to *published* specifications. • How can we help you? Send us the details of your coating problem.

PITT CHEM® TAR BASE ENAMELS

- Standard Grade
- Modified Grade
- Plasticized Grade
- Hotline
- Cold Applied Tar Base Coatings

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